SUPPORT FOR THE EPA NATIONAL MONITORING PROGRAMS

(UATMP, NATTS, CSATAM, PAMS, and NMOC Support)

Contract No. EP-D-14-030

2018

Quality Assurance Project Plan
Category 1

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2018 Quality Assurance Project Plan, Category 1
UATMP, NATTS, CSATAM, PAMS, and NMOC Support (Contract No. EP-D-14-030)

Approved by:

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U.S. EPA QA Manager: ______________________ Date: 7/26/18

U.S. EPA Delivery Order Manager: ______________________ Date: 7/26/18

ERG Program Manager: Julie C. Swift Date: 7/27/18

ERG Deputy Program Manager: Laura VanEnwyck Date: 7/27/18

ERG Program QA Officer: Donna Tedder Date: 7/27/18

ERG Deputy Program QA Officer: Jennifer Nash Date: 7/27/18

DISCLAIMER

This Category 1 Quality Assurance Project Plan has been prepared specifically to address the operation and management of the U.S. EPA National Monitoring Programs (UATMP, NATTS, CSATAM, PAMS and NMOC). The contents have been prepared in accordance with Level I Specifications of the EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5 and the EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5.

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SYMBOLS AND ABBREVIATIONS

AAC	Atmospheric Analysis and Consulting
AMTIC	Ambient Air Monitoring Technical Information Center
AQS	Air Quality Subsystem
ASTM	American Society for Testing and Materials
BFB	4-Bromofluorobenzene
BLK	Blank
BS/BSD	Blank Spike/Blank Spike Duplicate
CAA	Clean Air Act
CAR	Corrective Action Report
CCB	Continuing calibration blank
CCV	Continuing calibration verification
CFR	Code of Federal Regulations
COC	Chain of Custody
CSATAM	Community Scale Air Toxics Ambient Monitoring
CV	Coefficient of Variation
DFTPP	Decafluorotriphenylphosphine
DNPH	2,4-Dinitrophenylhydrazine
DPR	Daily Performance Check
DQOs	Data Quality Objectives
DUP	Duplicate
DVD	Digital Versatile Disk
EPA	U.S. Environmental Protection Agency
ERG	Eastern Research Group, Inc.
FACA	Federal Advisory Committee Act
FB	Field Blank
FC-43	perfluorotributylamine
FEM	Federal Equivalency Method
FID	Flame Ionization Detector
GC	Gas Chromatograph
GPRA	Government Performance and Results Act
HAPs	Hazardous Air Pollutant(s)
He	Helium
H_2	Hydrogen

SYMBOLS AND ABBREVIATIONS (Continued)

Hg	Mercury
HPLC	High Performance Liquid Chromatography
HSV	High standard verification
IC	Ion Chromatography
IC	Initial Calibration Standards (for ICP-MS)
ICAL	Initial Calibration
ICB	Initial Calibration Blank
ICP-MS	Inductively Coupled Plasma/Mass Spectrometer
ICSA/IFA	Interference Check Standard A
ICSAB/IFB	Interference Check Standard B
ICV	Initial calibration verification
ID	Identification
IS (or ISTD)	Internal Standard
KED	Kinetic Energy Discrimination
LCS	Laboratory Control Standard
LCV	Low Calibration Verification
LIMS	Laboratory Information Management System
LOQ	Limit of Quantitation
LRB	Laboratory Reagent Blank
m	Meter(s)
MB	Method Blank
MDLs	Method Detection Limit(s)
mL	Milliliter
mm	Millimeter
mM	Millimolar
MQOs	Measurement Quality Objective
MS	Mass Spectrometer
MS/MSD	Matrix Spike/Matrix Spike Duplicate
MUR	Method Update Rule
µg	Micrograms
µg/mL	Micrograms per milliliter
$\mu g/m^3$	Microgram per cubic meter
µL	Microliters
µm	Micrometer

SYMBOLS AND ABBREVIATIONS (Continued)

µg/mL	Micrograms per milliliter
N_2	Nitrogen
NAAQS	National Ambient Air Quality Standard
NATTS	National Ambient Toxics Trends Stations
NELAC	National Environmental Laboratory Accreditation Conference
NELAP	National Environmental Laboratory Accreditation Program
NIST	National Institute of Standards and Technology
NIOSH	National Institute for Occupational Safety and Health
ng	Nanogram
ng/m^3	Nanogram per cubic meter
nm	Nanometer
NMOC	Nonmethane Organic Compounds
NMP	National Monitoring Program
NO_x	Oxides of Nitrogen
O_3	Ozone
OAQPS	Office of Air Quality Planning and Standards
OD	Outer Diameter
OSHA	Occupational Safety and Health Administration
PAHs	Polycyclic Aromatic Hydrocarbons
PAMS	Photochemical Assessment Monitoring Stations
PCBs	Polychlorinated biphenyls
PDF	Portable Document Format
PDFID	Preconcentration Direct Flame Ionization Detection
PDS	Post digestion spike
PE	Performance Evaluation
POC	Parameter Occurrence Code
ppbC	Parts per Billion as Carbon
ppbv	Parts per Billion by volume
ppmC	Parts per Million as Carbon
psig	Pounds per square inch gauge
PT	Proficiency Testing
PUF	Polyurethane Foam
QA	Quality Assurance
QAPPs	Quality Assurance Project Plan(s)

SYMBOLS AND ABBREVIATIONS (Continued)

QC	Quality Control
QL	Quantitation Limit
RE	Relative Error
RF	Response Factor
RPD	Relative Percent Difference
RRF	Relative Response Factor
RRTs	Relative Retention Times
RSD	Relative Standard Deviation
RT	Retention Time
RTP	Research Triangle Park
SB	Solvent Blank
SIM	Selected Ion Monitoring
SIP	State Implementation Plan
SNMOC	Speciated Nonmethane Organic Compounds
SOPs	Standard Operating Procedure(s)
SQL	Sample Quantitation Limit
SRD	Serial dilution
SRM	Standard Reference Material
SSQC	Second Source Quality Control
STI	Sonoma Technology, Inc.
SVOC	Semivolatile Organic Compounds
TAD	Technical Assistance Document.
TSAs	Technical System Audits
TSP	Total Suspended Particulate
UAM	Urban Airshed Model
UATMP	Urban Air Toxics Monitoring Program
UPS	United Parcel Service of America
UV	Ultraviolet
VOCs	Volatile Organic Compound

DISTRIBUTION LIST

Copies of this plan and all revisions will be provided to:

- Jeff Yane, Work Assignment Manager, U.S. EPA, C404-02, RTP, NC
- Dave Shelow, Delivery Order Manager, U.S. EPA, C339-02, RTP, NC
- Greg Noah, AT QA Coordinator, U.S. EPA, C304-06, RTP, NC

U.S. EPA Regional contacts may obtain a copy of the QAPP by contacting the ERG Program Manager. It is the responsibility of each Regional contact to make copies of the plan for appropriate State personnel or to refer them to ERG Program Manager. The ERG staff working on this contract will receive a copy of this QAPP and all revisions.

PROJECT MANAGEMENT
SECTION 1
PROJECT/TASK ORGANIZATION

1.1 Assignment of Program Personnel

Table 1-1 presents the program organization listing the program assignment and responsible person for each aspect of the Environmental Protection Agency (EPA) National Monitoring Programs (NMP). The program organizational chart is presented in Figure 1-1. All Eastern Research Group, Inc. (ERG) staff working on this contract are provided access to a current electronic copy of this signed, EPA approved Quality Assurance Project Plan (QAPP).

ERG's primary support on this contract includes Nonmethane Organic Compounds (NMOC), Speciated Nonmethane Organic Compounds (SNMOC), Volatile Organic Compounds (VOCs), Polycyclic Aromatic Hydrocarbons (PAHs), Metals, Hexavalent Chromium, and other Hazardous Air Pollutants (HAPs). Subcontracting services are extended by ChromIan for onsite technical assistance for Photochemical Assessment Monitoring Stations (PAMS) analysis, Sonoma Technology, Inc. (STI) for data validation, Atmospheric Analysis and Consulting, Inc. (AAC) Lab for VOCs by Method TO-17, pesticides/Polychlorinated biphenyls (PCBs), anions, diisocyanates, and 4,4'-methylenedianiline, and RTI International for metals analysis, in the event of a large workload.

ERG is responsible to the client for the work of the subcontractor and choosing subcontractors that meet the applicable requirements for the methods and contracts. The subcontractor should meet the Data Quality Objectives (DQOs) requirements for the appropriate method. ERG shall maintain a record of subcontractor compliance, including documentation of subcontractor's Method Detection Limits (MDLs), QAPPs, etc. Sample analysis will not begin with the subcontractor until MDLs, QAPPs, etc., have been approved by EPA and ERG. Before sample analysis, the subcontractor may perform Proficiency Testing (PT) samples and/or Technical System Audits (TSAs) if they are available through Office of Air Quality Planning and Standards (OAQPS). If such measures are not available, ERG will request audit reports performed with the subcontract lab and will supply PT audits if requested by the EPA when analysis is contracted with the laboratory.

1.1.1 Program Manager

Ms. Julie Swift, an ERG Vice President, serves as the Program Manager for EPA's NMP. In this role, she has the primary responsibility for understanding program level needs, both EPA's and their clients' (i.e., State, Local, and Tribal agencies). Ms. Swift is ultimately accountable for providing timely, cost effective, and high-quality services that meet the needs of the NMP efforts. Her responsibility is ensuring EPA/client satisfaction by verifying that all components necessary for effective management are in place and active during the contract performance period. Ms. Swift coordinates with the ERG Quality Assurance (QA) Officer, and task leaders to provide EPA/client perspective, communicate technical issues and needs, and ensure the program staff facilitates decisions appropriate to their roles on Contract EP-D-14-030. She prepares budgetary and schedule information and prepares all information for presentation to EPA at scheduled program meetings. As the Program Manager, Ms. Julie Swift is responsible for the technical operation and the quality of the program on a day-to-day basis. She leads the analytical tasks and provides technical direction and support. She assists in the resolution of technical issues and serves as a resource for Task Leaders regarding any project issues. Ms. Swift also performs an overall review of the data that is reported monthly.

1.1.2 Deputy Program Manager

As the Deputy Program Manager, Ms. Laura Van Enwyck assists the Program Manager for EPA's NMP. She assists the Program Manager in all aspects of the technical operation and the quality of the program on a day-to-day basis. She assists the analytical Task Leaders and provides technical direction and support. She assists in the resolution of technical issues and serves as a resource for Task Leaders regarding project issues. Ms. Van Enwyck is also the Carbonyl and HAPs Support Task Leader.

1.1.3 Program Technical Adviser

The Program Technical Adviser, Mr. Dave Dayton assists in the resolution of technical issues. He communicates with ERG management and the technical staff for discussion of real and potential technical problems. He peer reviews draft and final program report products and provides oversight of efforts to evaluate and characterize data.

1.1.4 Program QA Coordinator

Ms. Donna Tedder, the Program and Laboratory QA Coordinator, is responsible for ensuring the overall integrity and quality of project results. Ms. Tedder, or her designee, will do a 10 percent QA review for all sample analyses delivered for reporting by the Program Manager. In the case of subcontracted work, 20 percent of data from subcontractor will be reviewed. The lines of communication between management, the Program QA Coordinator, and the technical staff are formally established and allow for discussion of real and potential problems, preventive actions, and corrective procedures. The key Quality Control (QC) responsibilities and QC review functions are summarized in Table 1-2. On major quality issues, Ms. Tedder reports independently to Ms. Jan Connery, ERG's corporate QA Officer.

1.1.5 Deputy Program QA Coordinator

The Deputy Program QA Coordinator, Ms. Jennifer Nash, is responsible for ensuring the integrity and quality of project results. The Deputy QA Coordinator will assist the Program QA Coordinator with the QA review for sample analyses delivered for reporting by the Program Manager. The major QC responsibilities and QC review functions are summarized in Table 1-2. The Deputy QA Coordinator will work closely with the Program QA Coordinator to ensure the overall quality of the Program.

1.1.6 Task Leaders

ERG Task Leaders are responsible for meeting the project objectives, meeting report schedules, and directing the technical staff in execution of the technical effort for their respective task(s). The Task Leaders will review 100 percent of all sample analyses. The Program QA Coordinator will request 10 percent of that data for review prior to data reporting by the Program Manager. The Task Leaders manage the day-to-day technical activities on delivery orders for this program. They assess and report on the project's progress and results (e.g., recordkeeping, data validation procedures, sample turnaround time) and ensure timely, high-quality services that meet the requirements in this QAPP.

Table 1-1
Program Organization

Program Assignment	Program Personnel Assigned	Phone Number	Email Address
Program Manager	Julie Swift	(919) 468-7924	julie.swift@erg.com
Deputy Program Manager	Laura Van Enwyck	(919) 468-7930	laura.vanenwyck@erg.com
Task Leader - Network Site Coordination	Randy Bower	(919) 468-7928	randy.bower@erg.com
Task Leader - Shipping and Receiving	Randy Bower	(919) 468-7928	randy.bower@erg.com
Task Leader - Air Toxics	Randy Bower	(919) 468-7928	randy.bower@erg.com
Task Leader - Carbonyl Analysis	Laura Van Enwyck	(919) 468-7930	laura.vanenwyck@erg.com
Task Leader – Hexavalent Chromium	Glenn Isom	(919) 468-7940	glenn.isom@erg.com
Task Leader – Metals	Randy Mercurio	(919) 468-7922	randy.mercurio@erg.com
Task Leader - NMOC Analysis	Mitchell Howell	(919) 468-7915	mitch.howell@erg.com
Task Leader - Semivolatiles	Scott Sholar	(919) 468-7951	scott.sholar@erg.com
Task Leader - SNMOC Analysis	Mitchell Howell	(919) 468-7915	mitch.howell@erg.com
Task Leader - PAMS Support *	Julie Swift	(919) 468-7924	julie.swift@erg.com
Task Leader - HAPs Support **	Laura Van Enwyck	(919) 468-7930	laura.vanenwyck@erg.com
Task Leader - Data Characterization	Regi Oommen	(919) 468-7829	regi.oommen@erg.com
Task Leader - Annual Report/AQS Entry	Jaime Hauser	(919) 468-7813	jaime.hauser@erg.com
Program Technical Adviser	Dave Dayton	(919) 468-7883	dave.dayton@erg.com
Program QA Coordinator	Donna Tedder	(919) 468-7921	donna.tedder@erg.com
Deputy QA Coordinator	Jennifer Nash	(919) 468-7881	jennifer.nash@erg.com
Project Administrator	Kerry Fountain	(919) 468-7962	kerry.fountain@erg.com

*Subcontracting support when requested from Chromian and Sonoma Technology, Inc.
**Subcontracting support when requested from AAC and RTI International (miscellaneous HAPs).

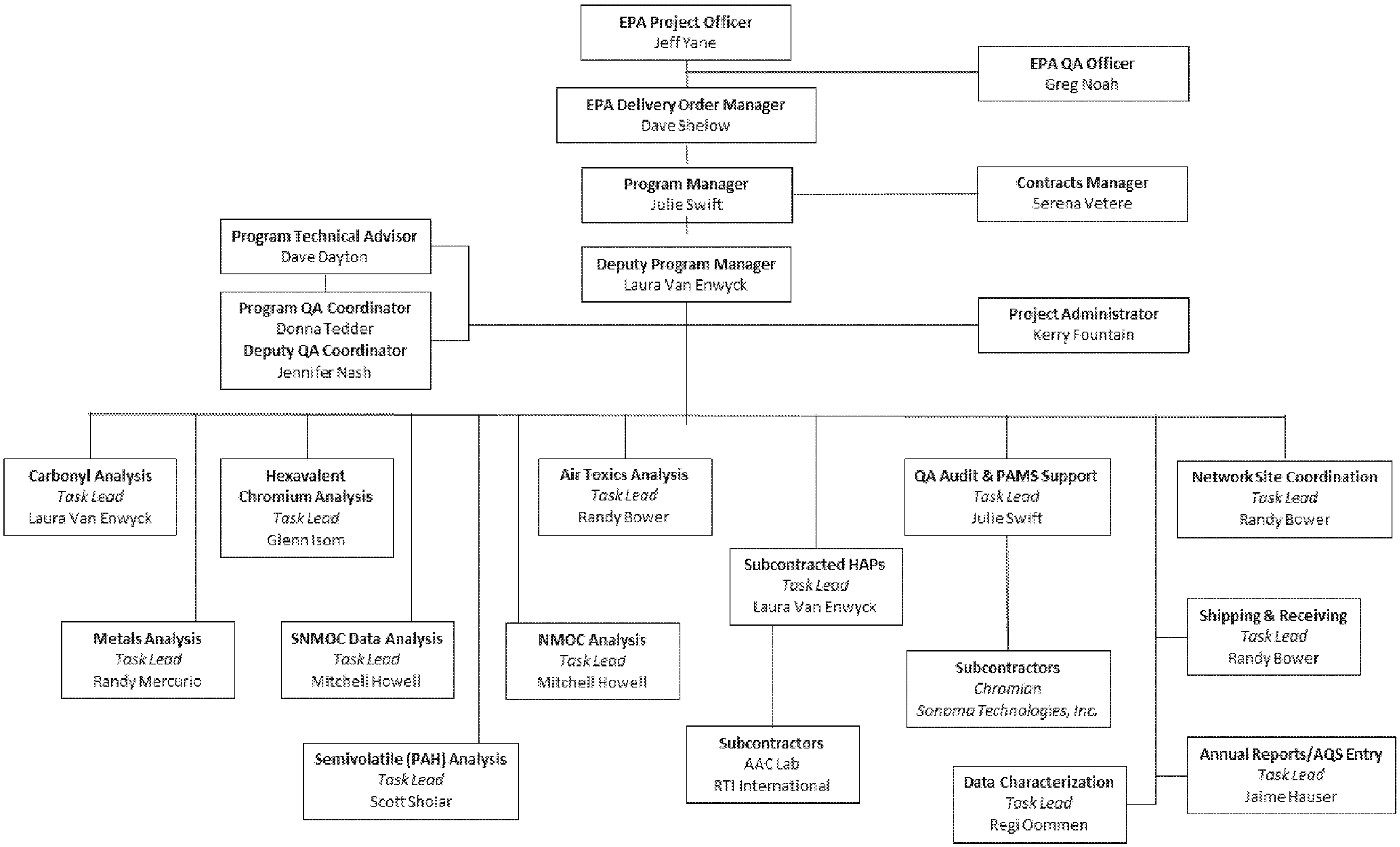


Figure 1-1. National Monitoring Programs Organizational Chart

Table 1-2
QC Responsibilities and Review Functions

Responsible Person	Major Responsibilities
Ms. Julie Swift, Program Manager	• Ensure overall timely performance of high quality technical services • Communicate technical issues and needs • Assist in the resolution of technical problems • Track all management systems and tools • Track deliverables and budget performance • Ensure appropriate level of staffing and committed resources exist to perform work • Communicate daily with the EPA/State/Local/Tribal agencies • Ensure data quality • Check information completeness • Review data completeness and quality before reporting to client • Review all reports • Report project performance (budget and deliverables) to EPA at scheduled meetings and in monthly progress reports • Day-to-day management of task leaders
Ms. Laura Van Enwyck, Deputy Program Manager	• Assist Program Manager where needed • Ensure overall timely performance of high quality technical services • Communicate technical issues and needs • Assist in the resolution of technical problems • Ensure appropriate level of staffing and committed resources exist to perform work • Communicate with the EPA/State/Local/Tribal agencies • Ensure data quality • Check information completeness • Review data completeness and quality before reporting to client • Day-to-day management of task leaders
Mr. Dave Dayton, Program Technical Adviser	• Assist in the resolution of technical problems • Communicate potential technical issues and needs • Review draft and final data reports
Ms. Donna Tedder, Program QA Coordinator	• Make QA recommendations • Review QAPP • Audit laboratory • Review QA reports • Evaluate the effect of technical issues on data quality • Review 10% of all data for reporting • Review documentation (SOPs, reports, etc.)

Table 1-2
QC Responsibilities and Review Functions (Continued)

Responsible Person	Major Responsibilities
Ms. Jennifer Nash, Deputy Program QA Coordinator	• Assist QA Coordinator where needed • Make QA recommendations • Review QAPP • Assist with laboratory audit(s) • Evaluate the effect of technical issues on data quality • Review 10% of all data for monthly reporting • Review documentation (SOPs, reports, etc.)
Task Leader(s)	• Review documentation • Review 100% of analytical data generated by analysts • Develop analytical procedures • Propose procedural changes • Train and supervise analysts • Meet task report schedules • Manage day-to-day technical activities • Check information completeness • Review instrument and maintenance log books • Review calibration factor drift • Perform preventive maintenance

SECTION 2

PROBLEM DEFINITION/BACKGROUND

The Clean Air Act (CAA) Amendments of 1990 required EPA OAQPS to set National Ambient Air Quality Standard (NAAQS) for the "criteria" pollutant ozone (O_3). In areas of the country where the NAAQS for O_3 was being exceeded, additional measurements of the ambient NMOC were needed to assist the affected States in developing/revising O_3 control strategies. Measurements of ambient NMOC are important to the control of VOCs that are precursors to atmospheric O_3. Due to previous difficulty in obtaining accurate NMOC concentration measurements, EPA started a monitoring and analytical program in 1984 to provide support to the States. ERG has continuously supported EPA for the NMOC programs since 1984.

In 1987, EPA developed the Urban Air Toxics Monitoring Program (UATMP) to help State, Local and Tribal air monitoring agencies characterize the nature and extent of potentially toxic air pollution in urban areas. Since 1987, several State and local agencies have participated in the UATMP by implementing ambient air monitoring programs. These efforts have helped to identify the toxic compounds most prevalent in the ambient air and indicate emissions sources that are likely to be contributing to elevated concentrations. Studies indicate that a potential for elevated cancer risk is associated with certain toxic compounds often found in ambient urban air(1). As a screening program, the UATMP also provides data input for models used by EPA, State, local and risk assessment personnel to assess risks posed by the presence of toxic compounds in urban areas. The UATMP program is a year-round sampling program, collecting 24-hour integrated ambient air samples at urban sites in the contiguous United States every 6 or 12 days.

The SNMOC program was initiated in 1991 in response to requests by State agencies for more detailed speciated hydrocarbon data for use in O_3 control strategies and Urban Airshed Model (UAM) input.

Title I, Section 182 of the CAA Amendments of 1990 requires States to establish PAMS as part of their State Implementation Plan (SIP) for O_3 nonattainment areas. The rule revises the ambient air quality surveillance regulations to include enhanced monitoring of O_3 and its precursors. The regulations promulgated in 1993 require monitoring of O_3, oxides of nitrogen (NO_X), selected carbonyl compounds, and VOCs. The required monitoring is complex and requires considerable lead time for the agencies to acquire the equipment and expertise to implement their PAMS network. Under the PAMS program, each site may require a different level of support with respect to sampling frequency, sampling equipment, analyses, and report preparation. Presampling, sampling, and analytical activities are performed according to the guidance provided in the Technical Assistance Document (TAD)[(2)], for Sampling and Analysis of Ozone Precursors, 1998 revision. The program objective of PAMS is to provide data that are consistent with the proposed rule for ambient air quality surveillance regulations in accordance with Code of Federal Regulations Title 40, Part 58 (40 CFR Part 58). The ERG team offers site support to any State that needs to set up a PAMS site and/or provide technical help. The specific analytical methodology applicable to the PAMS program will be discussed in this QAPP.

In 1999, EPA expanded this program to provide measurements of additional CAA HAPs to support the Government Performance and Results Act (GPRA). As required under the GPRA, EPA developed a Strategic Plan that includes a goal for Clean Air. Under this goal, there is an objective to improve air quality and reduce air toxics emissions to levels 75 percent below 1993 levels by 2010 in order to reduce the risk to Americans of cancer and other serious adverse health effects caused by airborne toxics.

In 2001, EPA designed a national network for monitoring air toxics compounds present in ambient air entitled the National Ambient Toxics Trends Station (NATTS). The primary purpose of the NATTS network is tracking trends in ambient air toxics levels to facilitate measuring progress toward emission and risk reduction goals. The monitoring network is intended for long term operation for the principle purpose of discerning national trends in air toxics ambient concentrations.

Beginning in 2003/2004, EPA conducted periodic Community Scale Air Toxics Ambient Monitoring (CSATAM) grant competitions. The resultant 1- to 2-year grants are designed to help State, Local, and Tribal communities identify and profile air toxics sources, characterize the degree and extent of local air toxics problems, and track progress of air toxics reduction activities. Grants have been awarded across the United States, in large, medium, and small communities. The ERG team can offer site support and analysis to any agency for the UATMP, NATTS and CSATAM programs.

The data obtained by following this QAPP will be used by EPA, State, Local, Tribal and risk assessment personnel to determine prevalent O_3 precursors and air toxics in the urban air. The data collected from the continuous yearly sites gives the data analyst consistent high quality analytical results. Sampling and analytical uncertainties are determined through this program by performing 10 percent sampling duplicate (or collocated) and analytical replicate samples for each of the ambient air sites.

This QAPP defines the preparation, sampling, laboratory analyses and QA/QC procedures conducted by ERG for EPA's NMP to deliver data of sufficient quality to meet the programs' objectives. Many of these procedures described in this QAPP are based on experiences obtained during previous National Program Studies.

SECTION 3
PROJECT/TASK DESCRIPTION

This section describes the activities performed under each of the major EPA NMP components (NMOC, SNMOC, UATMP, CSATAM, NATTS, and PAMS). ERG dedicates passivated canisters, sampling equipment and expendable sampling media to the program to maintain known quality that meets the program objectives. An applicable measurement methods list is presented in Table 3-1. Sampling and analysis are determined when delivery orders are provided by EPA.

3.1 PAMS, NMOC and SNMOC

The program objective of PAMS is to provide data that are consistent with the proposed rule for Ambient Air Quality Surveillance in accordance with 40 CFR Part 58. The ERG team can offer site support to any State that needs to set up a PAMS site and/or maintain it with technical help. Canister and/or carbonyl samples are collected typically every 3 days by State/Local/or Tribal agency personnel starting on the first of June through the end of September at each of the designated sites.

The NMOC and SNMOC programs require collection of ambient air samples over a 3-hour period. This sample collection period occurs from 6:00 - 9:00 a.m. local time to capture mobile source pollutants during the morning "rush hour" simultaneously with sunrise, which provides the energy necessary for many photochemical reactions. Weekday sampling will be the responsibility of the individual States involved in this program. Canister and/or carbonyl samples are collected by State/Local/or Tribal agency personnel every weekday, typically starting on the first Monday of June through the end of September at each of the designated sites.

ERG can provide sampler, sampler training, and any technical assistance needed throughout the monitoring program. At least one week before each sample collection episode, ERG ships the necessary clean, certified canisters and/or carbonyl cartridges to the site along with the field chain of custody (COC) forms. The time-integrated ambient samples are then collected and shipped to ERG for analysis.

3.2 UATMP, NATTS and CSATAM

The UATMP program was initiated as an analytical/technical support program focused on ascertaining ambient air levels of organic toxic species. The program has since expanded to provide for the measurement of additional HAPs and the standard sample collection frequency was increased to 1 in 6 days, with some sites continuing at 1 in 12 days.

The NATTS Network is intended for long term operation for the principle purpose of discerning national trends. The primary purpose of the NATTS network is tracking trends in ambient air toxics levels to facilitate measuring progress toward emission and risk reduction goals. The monitoring network is intended to be able to detect a 15 percent difference (trend) between two successive 3-year annual mean concentrations within acceptable levels of decision error. The standard sample collection frequency is 1 in 6 days.

The program objective of the CSATAM Program is designed to help State, Local, and Tribal communities identify and profile air toxics sources, characterize the degree and extent of local air toxics problems, and track progress of air toxics reduction activities. Grants have been awarded across the entire United States, in large, medium, and small communities. Awarded grants fall into one of three categories: community-scale monitoring, method development/evaluation, and analysis of existing data. The sample collection frequency may be 1 in 6 days or 1 in 12 days. Targeted pollutants generally reflect the NATTS core compounds, criteria pollutants, and/or pollutants related to diesel particulate matter.

The ERG team can offer site support and analysis to any State that needs VOC, carbonyl, or other analyses for the PAMS, UATMP, NATTS and CSATAM programs, as shown in Table 3-1. Relevant Standard Operating Procedures (SOPs) are also referenced in the table.

Table 3-1
List of Analytical and Support Services

Analysis	Based on Method	SOP (ERG-MOR-XXX)
Analysis		
Total NMOC	TO-12[3]	-060
Speciated NMOC/PAMS Hydrocarbons via GC/FID	TAD for Ozone Precursors[2]	-005
VOCs via GC/MS	TO-15[4]	-005
Concurrent SNMOC and VOC via GC/MS/FID	TAD for Ozone Precursors[2]/TO-15[4]	-005
Carbonyls via HPLC	TO-11A[5]	-024
PM_{10} HAP Metals via ICP-MS	IO-3.5[6]/EQL-0512-201[7]/ EQL-0512-202[8]	-095
TSP Hexavalent Chromium via IC	ASTM D7614[9]	-063
SVOC analysis via GC/MS (SCAN)	TO-13A[10] / Method 8270D[11]	-044***
PAH analysis via GC/MS (SIM)	TO-13A[10] / ASTM D6209-13[12]	-049
PCB/Pesticides via GC *	TO-4A[13]	*
Anions via IC *	NIOSH 7903[14] **	*
VOCs via GC/MS (from cartridge) *	TO-17[15]	*
Diisocyanates *	OSHA Method 42[16]	*
4,4'-Methylenedianiline *	NIOSH Method 5029[17]	*
Site Support		
NMOC/SNMOC	TAD for Ozone Precursors[2]	-046***
VOC	TO-15[4]	-003 or -021
Carbonyls	TO-11A[5]	-003 or -047
Hexavalent Chromium	ASTM D7614-12[9]	-013
PAMS Technical	NA	NA
PAMS QA	NA	NA
Other Services		
Performance Samples for VOC	TO-15[4]	-061
Performance Samples for Carbonyls	TO-11A[5]	-024
Performance Samples for PAH	TO-13A[10] / ASTM D6209-13[12]	-049
Performance Samples for PM10 HAP Metals	IO-3.5[6]/EQL-0512-201[7]/ EQL-0512-202[8]	-095
Performance Samples for TSP Hexavalent Chromium	ASTM D7614-12[9]	-063
Sampler Certification for Carbonyls	TO-11A[5]	-100
Sampler Certification for VOC	TO-15[4]	-030
Uniform Calibration Standards	TO-15[4]	-061
AQS Data Entry (per pollutant group)	NA	-098
Report Development/Data Characterization	NA	NA

*Will be supplied by subcontractor when analysis is requested.

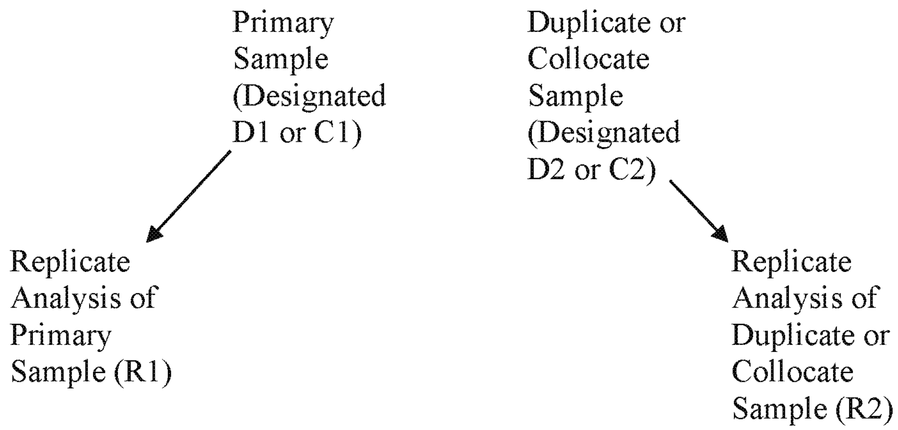
**NIOSH Method 7903 was replaced with 7906, 7907 and 7908.

***SOP is currently archived but will be updated if needed for sample analysis.

ERG can provide sampler, sampler training, and any technical assistance needed throughout the monitoring program. Canister and/or carbonyl samples are collected by State/Local/or Tribal agency personnel every 6 or 12-days at each of the designated sites. At least one week before each sample collection episode, ERG ships the necessary clean, certified canisters and/or carbonyl cartridges to the site along with the field COC forms. The time-integrated ambient samples are then collected and shipped to ERG for analysis.

ERG then prepares the program data for a final annual report describing sampling and analysis procedures, results, discussion of results, compilation of statistics, and recommendations. To determine the overall precision of analysis for the programs, replicate analyses (10 percent of the total number of samples) are used following the schematic shown in Figure 3-1. After the final data report receives approval by the EPA Project Officer and Delivery Order Manager, ERG distributes the final report to designated recipients. ERG provides the final data summaries to the associated agencies electronically in Excel® and Adobe® formats. ERG staff finalizes and uploads the data into the Air Quality Subsystem (AQS) database.

Figure 3-1. Duplicate/Collocate and Replicate Analysis Schematic



SECTION 4

DATA QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

As ERG performs measurement services only, DQOs for defining a toxics network program are not identified in this QAPP. A well-prepared description of the Measurements Quality Objectives (MQOs) can be found in the TAD for the NATTS Program prepared for EPA in October 2016[(18)]. This section will discuss the MQOs of the ERG laboratory analyses, emphasizing the levels of uncertainty the decision maker is willing to allow/accept from the analytical results. The DQOs for the four programs – NMOC, UATMP, PAMS, and CSATAM – are similar but are not identical. Therefore, the programs are discussed separately.

The NATTS TAD presents the requirements for collecting and reporting data for the NATTS network. Eighteen compounds have been identified as major risk drivers based on a relative ranking performed by EPA and have been designated as NATTS Core or "Tier I" compounds. All other reported compounds, for any NMP, are considered compounds of interest, but do not necessitate the NATTS MQOs. The Tier I compounds are acknowledged throughout this document. ERG exemptions from the NATTS TAD are listed in Appendix A.

Once a DQO is established, the quality of the data must be evaluated and controlled to ensure that data quality is maintained within the established acceptance criteria. MQOs are designed to evaluate and control various phases (sampling, preparation, analysis) of the measurement process to ensure that the total measurement uncertainty is within the range prescribed by the DQOs. MQOs can be defined in terms of the following data quality indicators:

<u>Precision</u> - a measure of mutual agreement between individual measurements performed according to identical protocols and procedures. This is the random component of error.

<u>Bias</u> - the systematic or persistent distortion of a measurement process that causes error in one direction. Bias is determined by estimating the positive and negative deviation from the true value as a percentage of the true value.

Representativeness - a measure of the degree to which data accurately and precisely represent a characteristic of population, parameter variations at a sampling point, a process condition, or an environmental condition.

Detectability - the determination of the low range critical value of a characteristic that a method-specific procedure can reliably discern.

Completeness - a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct, normal conditions. Data completeness requirements are included in the reference methods (see References, Section 21).

Comparability - a measure of the level of confidence with which one data set can be compared to another.

Bias has been the term frequently used to represent closeness to "truth" and includes a combination of precision and bias error components. The MQOs listed will attempt to separate measurement uncertainties into precision and bias components. Table 4-1 lists the MQOs for pollutants to be measured in all areas of the UATMP, NATTS, CSATAM, PAMS, and NMOC program.

Analytical Precision is calculated by comparing the differences between Replicate analyses (two analyses of the same sample) from the arithmetic mean of the two results as shown below. Replicate analyses with low variability have a lower Relative Percent Difference (RPD) (better precision), whereas high variability samples have a higher RPD (poorer precision).

$$RPD = \frac{|X_1 - X_2|}{\bar{X}} x\ 100$$

Where:

X_1 = Ambient air concentration of a given compound measured in one sample;
X_2 = Concentration of the same compound measured during replicate analysis;
$\bar{X}$ = Arithmetic mean of X_1 and X_2.

Method precision is calculated by comparing the concentrations of the duplicates/collocates for each pollutant. The Coefficient of Variation (CV) calculation shown below is ideal when comparing paired values, such as a primary concentration versus a duplicate concentration.

$$CV = 100 \times \sqrt{\frac{\sum_{i=1}^{n} \left[\frac{(p-r)}{0.5 \times (p+r)}\right]^2}{2n}}$$

Where:

p = the primary result from a duplicate or collocated pair;
r = the secondary result from a duplicate or collocated pair;
n = the number of valid data pairs (the 2 adjusts for the fact that there are two values with error).

Table 4-1
Measurement Quality Objectives for the National Program (UATMP, NATTS, CSATAM, PAMS, NMOC)

Program	Reporting Units	Precision from analysis of Replicate Samples (RPD)	Precision (CV) from collection of Duplicate/Collocate Samples	Representativeness	Comparability/ Based on Method	Bias	Completeness	Minimum Detection Limits*
NMOC	ppmC	≤ 10%	≤ 20%	Neighborhood	GC-PDFID EPA Compendium Method TO-12(3)	± 25%	>85%	To be determined upon need
SNMOC	ppbC	≤ 25% ≥ 5x MDL	≤ 25% ≥ 5x MDL	Neighborhood	GC-FID TAD for O_3 Precursors (2)	± 25%	>85%	See Table 11-12
VOC	ppbv	≤ 25% ≥ 5x MDL	For NATTS Tier I compounds, ≤15%, others ≤ 25% ≥ 5x MDL	Neighborhood	GC-FID/MS EPA Compendium Method TO-15(4)	± 25%	>85%	For NATTS Tier I, see NATTS TAD Table 4.1-1 Others, see Table 11-13
Carbonyls	ppbv	≤ 10% ≥ 0.5 µg/cartridge	For NATTS Tier I compounds, ≤15%, others ≤ 20% ≥ 0.5 µg/cartridge	Neighborhood	HPLC EPA Compendium Method TO-11A(5)	± 25%	>85%	For NATTS Tier I, see NATTS TAD Table 4.1-1 Others, see Table 11-14
Metals	ng/ per cubic meter (ng/m^3)	≤ 20% ≥ 5x MDL	For NATTS Tier I compounds, ≤15%, others ≤ 20% ≥ 5x MDL	Neighborhood	ICPMS IO-3.5(6)/EQL-0512-201(7)/ EQL-0512-202(8)	± 25%	>85%	For NATTS Tier I, see NATTS TAD Table 4.1-1 Others, see Table 11-16
Hexavalent Chromium	ng/m^3	≤ 20% for conc. > 5x MDL	≤ 20%	Neighborhood	IC-UV Detector ASTM D7614-12(9)	± 25%	>85%	0.0038 ng/m^3

*For NATTS Tier 1 compounds, minimum detection limits are listed in the NATTS TAD.

Table 4-1
Measurement Quality Objectives for the National Program (UATMP, NATTS, CSATAM, PAMS, NMOC) (Continued)

Program	Reporting Units	Precision from analysis of Replicate Samples (RPD)	Precision (CV) from collection of Duplicate/Collocate Samples	Representativeness	Comparability/ Based on Method	Bias	Completeness	Minimum Detection Limits
Semivolatiles	micro-gram/m^3 ($\mu g/m^3$)	≤ 10% for conc. ≥ 0.5 μg/mL	For NATTS Tier I compounds, ≤15%, others ≤ 20% for conc. ≥ 0.5 μg/mL	Neighborhood	GC/MS EPA Compendium Method TO-13A(10) and ASTM D6209-13(12), (or SW-846 Method 8270D(11))	± 25%	>85%	For NATTS Tier I, see NATTS TAD Table 4.1-1 Others, see Table 11-15
PCB/ Pesticides	ng/m^3	≤ 15%	≤ 15%	Neighborhood	GC EPA Compendium Method TO-4A(13)	± 25%	>85%	To be determined upon need
Anions	ppbv	≤ 15%	≤ 15%	Neighborhood	IC NIOSH Method 7903(14)	± 25%	>85%	To be determined upon need
VOCs via cartridge	ppbv	≤ 15%	≤ 15%	Neighborhood	GC/MS EPA Compendium Method TO-17(15)	± 25%	>85%	To be determined upon need
Diisocyanates	$\mu g/m^3$	≤ 15%	≤ 15%	Neighborhood	HPLC OSHA Method 42(16)	± 25%	>85%	To be determined upon need
4,4'-Methylene-dianiline	$\mu g/m^3$	≤ 15%	≤ 15%	Neighborhood	HPLC NIOSH Method 5029(17)	± 25%	>85%	To be determined upon need

*For NATTS Tier 1 compounds, minimum detection limits are listed in the NATTS TAD.

SECTION 5
SPECIAL TRAINING REQUIREMENTS/CERTIFICATION

The activities of EPA's NMP are performed using accepted EPA, National Institute for Occupational Safety and Health (NIOSH), and Occupational Safety and Health Administration (OSHA) sampling and analytical protocols for the field sampling training personnel and analytical laboratory staff.

5.1 Field Activities Training Personnel

Field activities training personnel involved in this project have over 30 years of experience in the duties they will be performing in the field. The training of ERG field activities personnel is recorded in the ERG Training Records files. Special certification is not needed for an operator to set up the sampling systems. Each State should document and record the training of their personnel on the field testing procedures provided by ERG.

The States' field testing staff will be subject to on-site surveillance by EPA. ERG's Task Leader will provide appropriate corrective action enforcement, if necessary, for the ERG personnel setting up the sampling equipment and the field testing staff. ERG provides on-the-job training in the field on sampler use and maintenance, for supervisors and field site operators. The appropriate SOPs used during training are presented in Appendix C. ERG does not provide SOPs for sampling systems that are not maintained by ERG. Sampling System Training forms used during operator training in the field is presented in Figure 7.2 for VOC/Carbonyl and Carbonyl samplers. The forms will only be provided when new site personnel are trained on the sampling systems. After training is completed and signed in the field, the yellow copy is retained for site records. The original copy is scanned in the laboratory and stored by the QA coordinator.

The sampling equipment for monitoring sites may be inside a sampling building or outside. There are no hazards inherent to the samplers and no special safety training or equipment will be required. Site hazards should be addressed on a site-by-site basis by the site operator's SOPs. All ERG field activities training personnel will follow the ERG Corporate Health and Safety Plan.

5.2 Analytical Laboratory Personnel

Analytical laboratory personnel involved in this project have been trained in their tasks and have up to 30 years of experience in the duties they will be performing in the analytical laboratory. Training of ERG laboratory personnel is recorded in ERG Training Records in an Excel® database and filed as a hardcopy. It is the responsibility of the trainee and the laboratory's Project Administrator to keep the Training Records up to date. It is the responsibility of the Program Manager and Quality Assurance Coordinator to approve analysis training records. Normal training and overview is provided to the analyst by the Task Leader for that analysis. Technical training includes general techniques and specific training based on the appropriate SOP, method, and program QAPP. The trainee first observes the task, then performs the task under supervision of the trainer, then performs the task under supervision of the Task Lead (if the Task Lead is not the trainer). After training, demonstration of each personnel's ability to perform an analytical task involves repeated measurements of a standard, which is described in more detail in each analytical SOP. Currently, no special certifications are needed for the analysis of the ambient samples received for these programs.

ERG maintains appropriate SOPs for each of the analytical methods. These SOPs are presented in Appendix C. All SOPs document equipment and/or procedures required to perform each specific laboratory activity. Laboratory staff will be subject to on-site surveillance by the QA staff and periodic performance evaluation (PE) samples. These audits will assure the program that the appropriate analysts and analytical procedures are being used. The samples involved in this program are generated by monitoring air emissions. Health and Safety training is performed annually. The laboratory personnel will adhere to the ERG Corporate Health and Safety manual.

SECTION 6
DOCUMENTATION AND RECORDS

The EPA NMP are a collection of individual ambient monitoring programs that generate documents and records that need to be retained/archived. All ERG staff working on this contract are provided access to a current electronic copy of this signed, EPA approved QAPP. Annually, the staff is required to sign a form to document that they read and understood the QAPP. In this QAPP, ERG's reporting package (information required to support the analytical results) includes all data required to be collected as well as support data deemed important by ERG/EPA.

6.1 Data Management

ERG has a structured records management system that allows for the efficient archive and retrieval of records. Each laboratory archives the data from the computer systems onto the shared network drive. The laboratory paper copies of all analyses are stored on site in a secured temperature-controlled area for up to five years after the close of the contract. The laboratory also archives the data in the Laboratory Information Management System (LIMS) data server which is backed up weekly, monthly, and biannually. The Program Manager has final authority for the storage, access to, and final disposal of all records kept for the EPA NMP.

6.2 Preliminary Monthly Data Reports

Preliminary monthly summary data reports are sent in Adobe Portable Document Format (PDF) and Excel formats to EPA and appropriate State/Local/Tribal agencies. The monthly data reports will include analytical results, associated MDL, final units, associated QC samples, and data qualifiers.

6.3 Quarterly QA Report

A QA report for each type of data analysis is sent to EPA and appropriate State/Local/Tribal agencies on a quarterly basis in the form of control charts including initial calibration verifications, continuing calibration verifications, method blanks, initial calibration blanks, continuing calibration blanks, and blank spikes.

6.4 Annual Summary Reports Submitted to EPA

Hard copies of the final report are presented to EPA contacts at the end of the sampling period. State/Local/Tribal agencies receive electronic copies (i.e., PDF). The final report is submitted for the data collected from January 1 to December 31 of the previous year. The report can contain the following information:

- Names of participating sites and corresponding metadata information, including city name, location and the AQS codes;
- Description of the sampling and analytical methodologies used by the laboratory;
- Completeness of the monitoring effort for each site;
- Background information on the methodology used to present and analyze the data;
- General combined and individual site summary of the year's results;
- Discussion of different trends for the select HAPs chosen for analysis;
- Risk screening evaluations using toxicity factors (e.g., UREs or RfCs);
- Variability analysis (intra-site and seasonal comparisons);
- Pollution roses to determine predominant direction for select compounds;
- Discussion of precision and accuracy and other prevalent QC concerns; and
- Yearly discussions of conclusions and recommendations.

If corrections are needed after the final report is presented to EPA, the report is easily retrieved, and corrections are sent to all relevant personnel.

6.5 Records and Supporting Data

All raw data required for the calculation of air toxics concentrations, submission to the EPA/AQS database, and QA/QC data are collected electronically or on data forms that are included in the field and analytical methods sections. All hardcopy information is filled out in indelible ink. Corrections are made by inserting one line through the incorrect entry, initialing the correction (ERG maintains a signature log), and placing the correct entry alongside the incorrect entry, if this can be accomplished legibly, or by providing the information on a new line. Table 6-1 presents the location of the data records for field and laboratory operations stored at the ERG laboratory.

Table 6-1. Data Documentation and Records

Item	Record	Short Term Location Storage	Long Term Location Storage
Field Operations			
Sampling System Training	Sampling System Training Form	ERG	Copy scanned and hardcopy stored by ERG
COC	ERG COCs	Field gets "pink" copy, ERG gets "yellow" and "white" copy	Copy scanned and stored on ERG LIMS
QC Sample Records (field blanks, duplicate/ collocated, sample integrity, etc.)	COC	Field	Copy scanned and stored on ERG LIMS
General Field Procedures	COC	Field	Copy scanned and stored on ERG LIMS
Laboratory Records			
Sample Prep Data	Bench sheets	Hardcopy filed, LIMS, shared network drive	Hardcopy archived, LIMS, shared network drive

Table 6-1. Data Documentation and Records, Continued

Item	Record	Short Term Location Storage	Long Term Location Storage
Laboratory Operations			
Sample Management Records (sample receipt, handling, storage, etc.)	COCs	LIMS, with sample analytical data	LIMS, with sample analytical data
Test Methods	SOPs	Hardcopy filed, shared network drive	Shared network drive
QA/QC Reports (General QC records, MDL information, calibration, etc.)	Individual records for each analysis	Hardcopy filed, shared network drive	Hardcopy archived, shared network drive
Corrective Action Reports	Individual records for each analysis	Hardcopy filed, a copy in data package if appropriate	All copies archived
Data Reduction, Verification, and Validation			
Electronic Data (used for reporting and AQS)	Excel® and Access®	Shared network drive	Shared network drive

6.5.1 Notebooks

ERG issues laboratory notebooks upon request. These notebooks are uniquely numbered and associated with the laboratory personnel. Notebooks are archived upon completion for at least 5 years from the end of a project. Although LIMS data entry forms are associated with all routine environmental data operations, the notebooks can be used to record additional information about these operations. The procedures for maintaining notebooks are presented in *SOP for Maintaining Laboratory Notebooks* (ERG-MOR-039) in Appendix C.

Field Notebooks - Field notebooks are the responsibility of EPA, States, Local or Tribal agencies as ERG is not responsible for the collection of samples.

Laboratory Notebooks - Notebooks are associated with general procedures such as calibration of analytical balances, standard preparation logs, etc., used in this program.

Logbooks are generated and bound by the laboratory's Project Administrator for procedures such refrigerator/freezer temperatures, canister cleaning, etc. Logbook pages have a unique version identifier. Upon completion, logbooks are archived indefinitely, at a minimum at least 5 years from the end of a project.

6.5.2 Electronic Data Collection

To reduce the potential for data entry errors, automated systems are utilized (where appropriate) and record the same information that is found on data entry forms. In order to provide a back-up, hardcopy data collected on an automated system will be stored for 5 years after the end of the closed EPA NMP contract.

6.6 Data Reporting Package Archiving and Retrieval

In general, all the information listed above will be retained for at least 5 years from the date of the end of the closed contract with EPA. However, if any litigation, claim, negotiation, audit, or other action involving the records has been started before the expiration of the 5-year period, the records will be retained until completion of the action and resolution of all issues which arise from it, or until the end of the regular 5-year period, whichever is later. The long-term storage is on-site in a locked climate-controlled file room with limited-access. The Project Administrator keeps a record of documents entering and leaving long-term storage. Access to the facility storage area is limited to authorized personnel only.

6.7 Quality System Document Control

To ensure the use of the most current version of quality system documents, all quality documents (QAPP, SOPs, etc.) generated at the ERG Laboratory must be uniquely identified. Original documents shall include the date of issue, revision number, page number, the total number of pages, and appropriate signatures. Copies of quality documents shall be controlled and include the date of issue, revision number, page number, the total number of pages, and copy control number. When an original quality document is updated, the QA Coordinator or designee will ensure that the copy documents are also updated, and old versions are destroyed. During the project, revised QAPPs will be circulated to appropriate EPA personnel and ERG's laboratory staff. For copies of documents out of the laboratory's control, a stamp or watermark stating "Uncontrolled" or "Draft", if applicable, will be applied. Each approved QAPP will be posted on EPA's Ambient Air Monitoring Technical Information Centers (AMTIC) Website without the associated SOPs.

MEASUREMENT DATA ACQUISITION
SECTION 7
SAMPLING PROCESS DESIGN

Sampling procedures for the NMOC, SNMOC, UATMP, NATTS, and CSATAM programs are discussed in this section. ERG provides site-specific support for the PAMS and HAPs sampling. All parameters listed in this section are necessary for the sampling systems listed below. ERG is not responsible for the collection of samples nor the design of these programs.

7.1 NMOC and SNMOC Canister Samplers

Sampling for NMOC and SNMOC takes place each workday from the beginning of June to the end of September at designated NMOC and SNMOC sites from 6:00 a.m. to 9:00 a.m. local time. Sampling procedures have been discussed in detail in other documents. [1, 2] Figure 7-1 is a diagram of the ERG sampling system used for collecting the ambient air samples. Clean, evacuated passivated stainless-steel canisters are shipped daily from ERG's Research Triangle Park (RTP) Laboratory to the NMOC and SNMOC sites. Canisters are connected to the sampling system by local operators. The digital timer automatically activates the pump and solenoid valve to start and stop sample collection. The pump pressurizes air samples during the sampling period to about 15 pounds per square inch gauge (psig), and the flow control valve (variable orifice) ensures a constant sampling rate over the 3-hour period. A 2-micron stainless steel filter is installed in the sampling line to remove particulate from the ambient air that may damage or plug the variable orifice. The sample probe inlet is positioned from 2 to 10 meters (m) above ground level.

ERG installs the sampling systems at the site location and trains associated local operators on site. Operator training is documented on the Sampler Training Form (Figure 7-2). It is the responsibility of the local operators to operate the sampling apparatus and complete the field sample COC form that ERG supplies with each canister. ERG staff maintain telephone

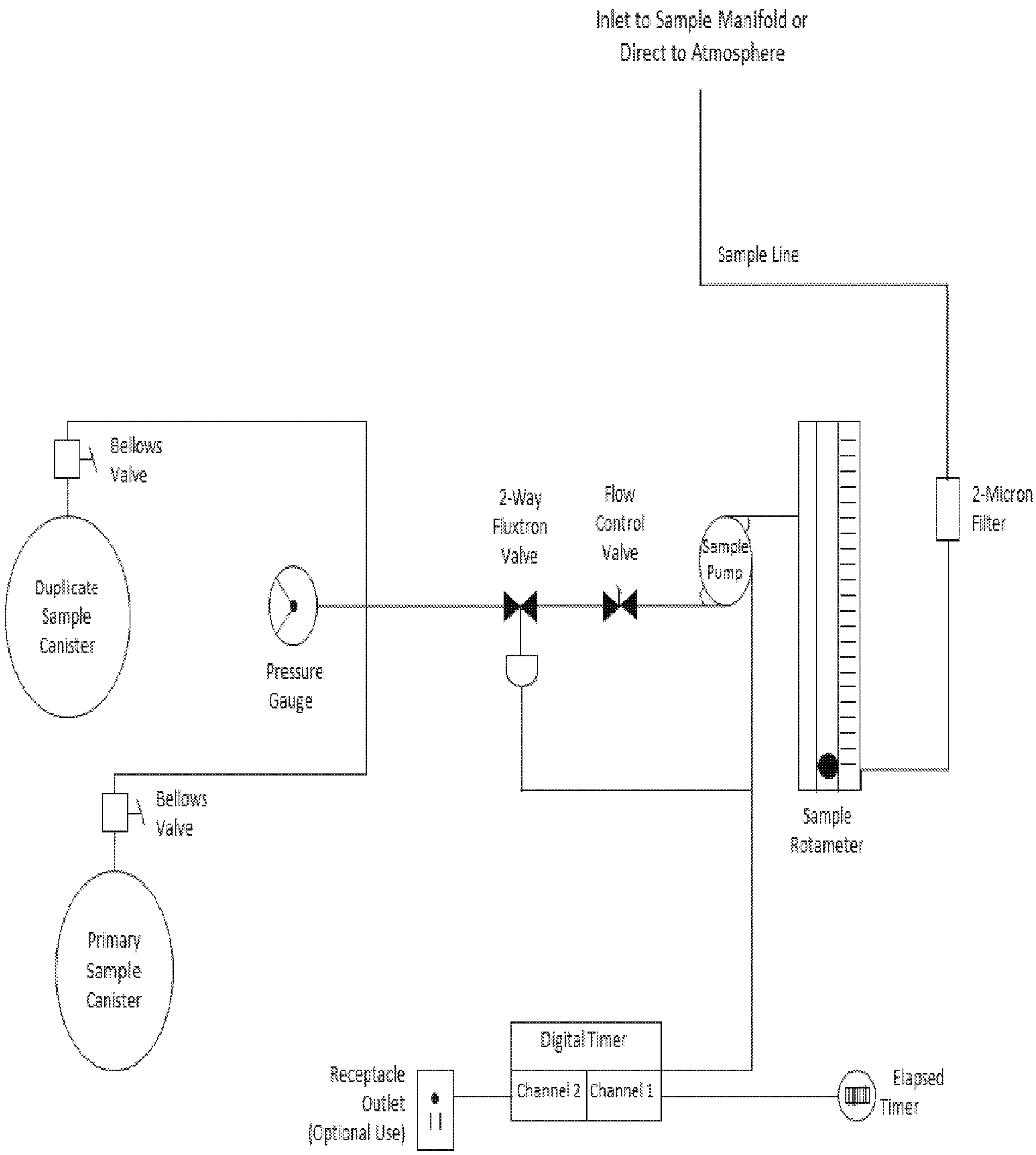


Figure 7-1. NMOC, SNMOC, and 3-Hour Air Toxics Sampling System Components

VOC/Carbonyl Sampling System Training

3-2018

Installation Date: ____________________

Trainer: ____________________

Site ID: ____________________

Copy of SOP on Site: (Y/N) ____________

Installed Sampler ID #: ____________________

Replaced Sampler ID #: ____________________

Time Set: ____________________

Carb Line Replaced: (Y/N) ____________

Timer Set: ____________________

VOC Line Replaced: (Y/N) ____________

Trainee:	Signature:	Date:

NOTES:

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ERG assumes no personal and/or property liability realized by the user from the use of ERG provided equipment. The user, by virtue of accepting the ERG equipment for use, undertakes any/all personal and/or property liabilities that could be associated with its use (including operational, housing, and/or safety).

Figure 7-2. VOC/Carbonyl Sampler Training Form

and/or email contact throughout the project to provide whatever assistance is needed to resolve technical issues that arise during the sampling program.

For a 3-hour ambient air sample, NMOC, SNMOC, and VOC measurements may all be performed from the same canister. Refer to Section 7.2 for sampler certification.

7.2 VOC and Carbonyl 24-Hour Samplers

ERG provides the sites with a sampling schedule each year. A total of 31 sampling days will be scheduled per site for a 12-day sampling schedule and 61 sampling days for the 6-day sampling schedule. Days for duplicate (or collocated) sampling will also be designated. The 2018 Sampling calendar is presented in Appendix B.

Prior to installation of an ERG sampler at a UATMP, NATTS or CSATAM site, the sampler is certified at the ERG laboratory. Certification establishes that the system is functioning correctly and provides for the appropriate level of specified compound recovery and cleanliness. To certify the sampling system, cleaned, humidified nitrogen (N_2) is first flushed through the sampler for at least 24 hours to remove the potential for organic contaminants in the system. The canister sub-system of the samplers is then challenged with a mixture of representative VOCs at known concentrations to qualify the sampler recovery characteristics (as recommended in the NATTS TAD)[18]. A Sampling System Blank is then collected in canisters and on carbonyl cartridges and is analyzed based on EPA Compendium Method TO-15[4] and Method TO-11A[5] to verify that the system meets the required cleanliness criteria and can produce non-biased samples (as required by the NATTS TAD[18]). These results are documented in a file specific to each sampler by system identification number. The certification procedures are presented in *SOP for Canister Sampling System Certification Procedures* (ERG-MOR-030) and *SOP for Carbonyl System Certification Procedures* (ERG-MOR-100) in Appendix C.

Integrated ambient air samples are collected in 6-liter passivated stainless-steel canisters (SUMMA, Silonite®, TO-Can, etc.) and carbonyl cartridges for a 24-hour period beginning at midnight for each scheduled sampling event. Carbonyl cartridges are shipped cold and the cleaned, quality-controlled canisters are shipped under vacuum to the site from the ERG laboratory. After sampling, the final pressure in the canister should ideally be between 2 to 8 inches of Mercury ("Hg) vacuum. The sampling assembly for the sample collection is shown in Figure 7-3.

The physical mechanism for filling the canister is vacuum displacement. The vacuum pump shown in Figure 7-3 is used to purge the mass flow controller and the sample inlet lines. A second vacuum pump is used to draw ambient air through the carbonyl sampling probe and cartridges. Ozone is removed from the sample stream prior to collection on the 2,4-Dinitrophenylhydrazine (DNPH) sampling cartridge. To accomplish O_3 removal, the sample stream (ambient air) is drawn through a potassium iodide-coated denuder O_3 scrubber which is an internally integrated component of the sampler. Carbonyl sampling can occur at sites at the same time as the canister samples are taken or on separate samplers.

7.3 Carbonyl Only 24-Hour Samplers

Carbonyl samples are collected using DNPH-impregnated sampling cartridges with an integrated sampling system (e.g., vacuum pump, capillary critical orifices, and O_3 scrubbers), shown in Figure 7-4. Ambient air is drawn through the cartridges via a separate sampling probe. A potassium iodide-coated denuder O_3 scrubber is an internally integrated component of the sampler that removes O_3 from the sample stream prior to the DNPH sampling cartridge.

Prior to installation of an ERG sampler at a UATMP, NATTS or CSATAM site, the sampler is certified at the ERG laboratory. Certification establishes that the system is functioning correctly and provides for the appropriate level of cleanliness. To certify the sampling system, cleaned, humidified N_2 is first flushed through the sampler for at least 12 hours to remove the potential contaminates from the system. A Sampling System Blank and a reference blank are then collected on carbonyl cartridges and are analyzed based on EPA

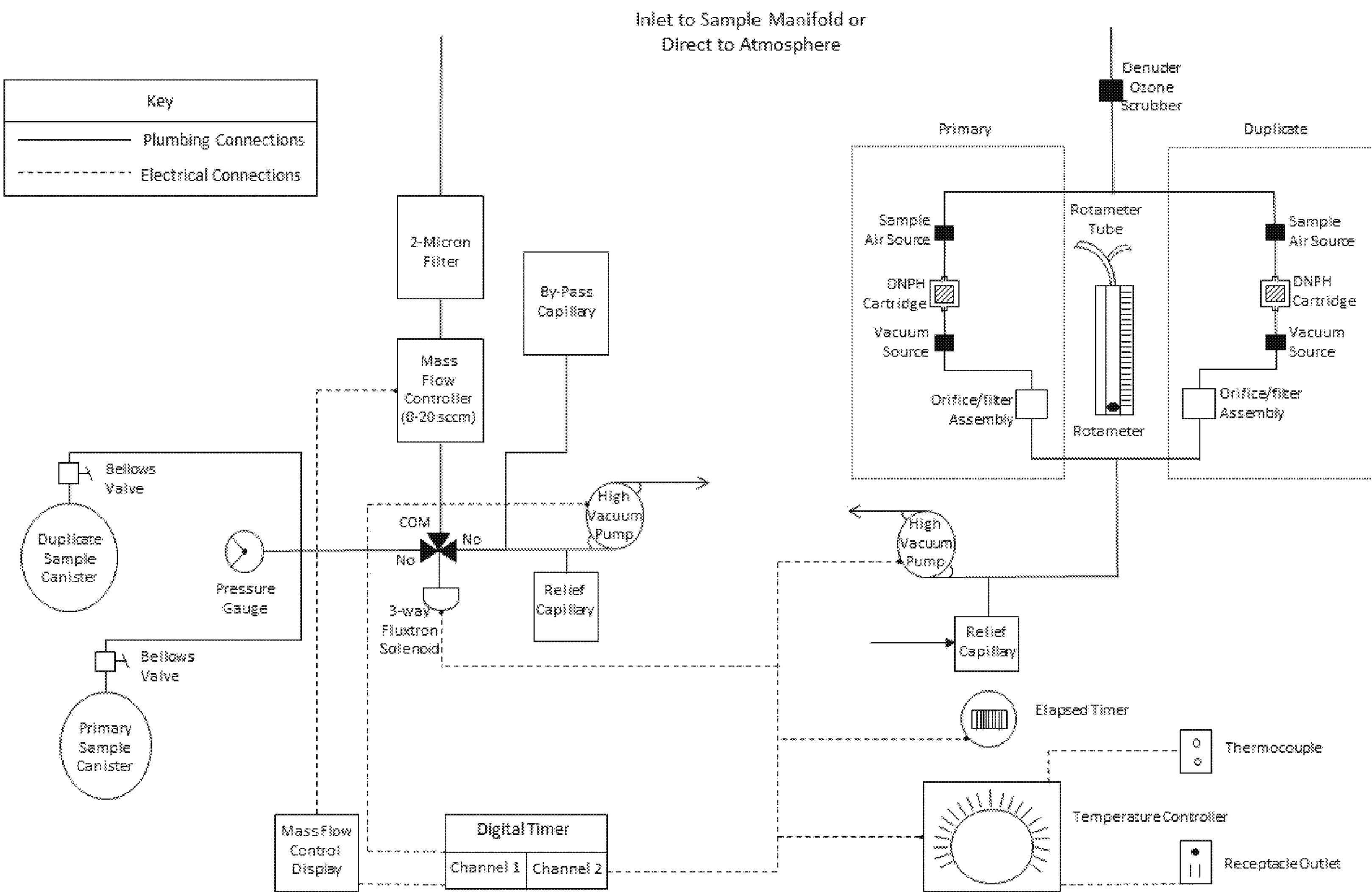

Figure 7-3. 24-Hour Integrated Air Toxics Sampling System Components

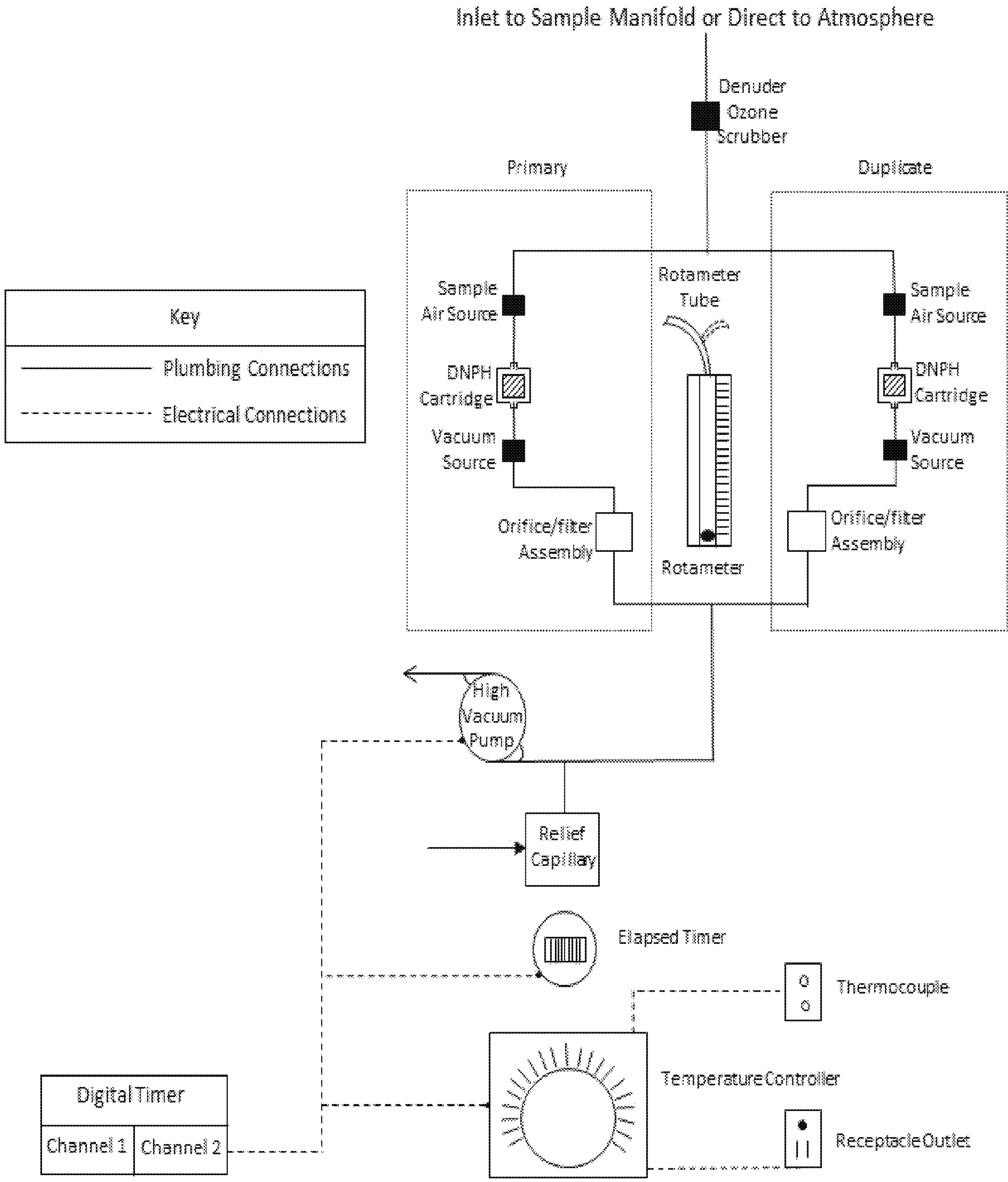

Figure 7- 4. Carbonyl Sampling System Components

Compendium Method TO-11A[5] to verify that the system meets the required cleanliness criteria and can produce non-biased samples as required by the NATTS TAD[18]. These results are documented in a permanent file specific to each sampler by system identification number. The certification procedure is presented in the *SOP for Carbonyl Sampling System Certification* (ERG-MOR-100) in Appendix C.

A total of 31 sampling cartridges for a 12-day sampling schedule and 61 sampling cartridges for a 6-day sampling schedule will be collected and analyzed per site. Duplicate (or collocated) samples and field blanks will be collected monthly and are designated in the 2018 Sampling calendar presented in Appendix B.

7.4 Hexavalent Chromium Samplers

Sodium bicarbonate-impregnated cellulose filters are connected to the Hexavalent Chromium sampler as shown in Figure 7-5 and ambient air is drawn through the filters through a glass sampling probe using Teflon sampling lines. Prepared filters are shipped to each site for the hexavalent chromium sampling. ERG ships the bicarbonate-impregnated sodium cellulose filters to each site in coolers (chilled with blue ice packs). The samples are collected for a 24-hour period. Disposable polyethylene gloves are used by the field operators when handling the filters to reduce background contamination. After sampling, the filters are removed from the sampling apparatus, sealed, and returned to the ERG laboratory in the coolers and ice packs in which they were received. Additional qualifying information for the hexavalent chromium sampling and analysis techniques is presented in the American Society for Testing and Materials (ASTM) D7614-12[9] method and specific details are provided in ERG's *SOP for the Preparation and Analysis of Ambient Air for Hexavalent Chromium by Ion Chromatography* (ERG-MOR-063) presented in Appendix C.

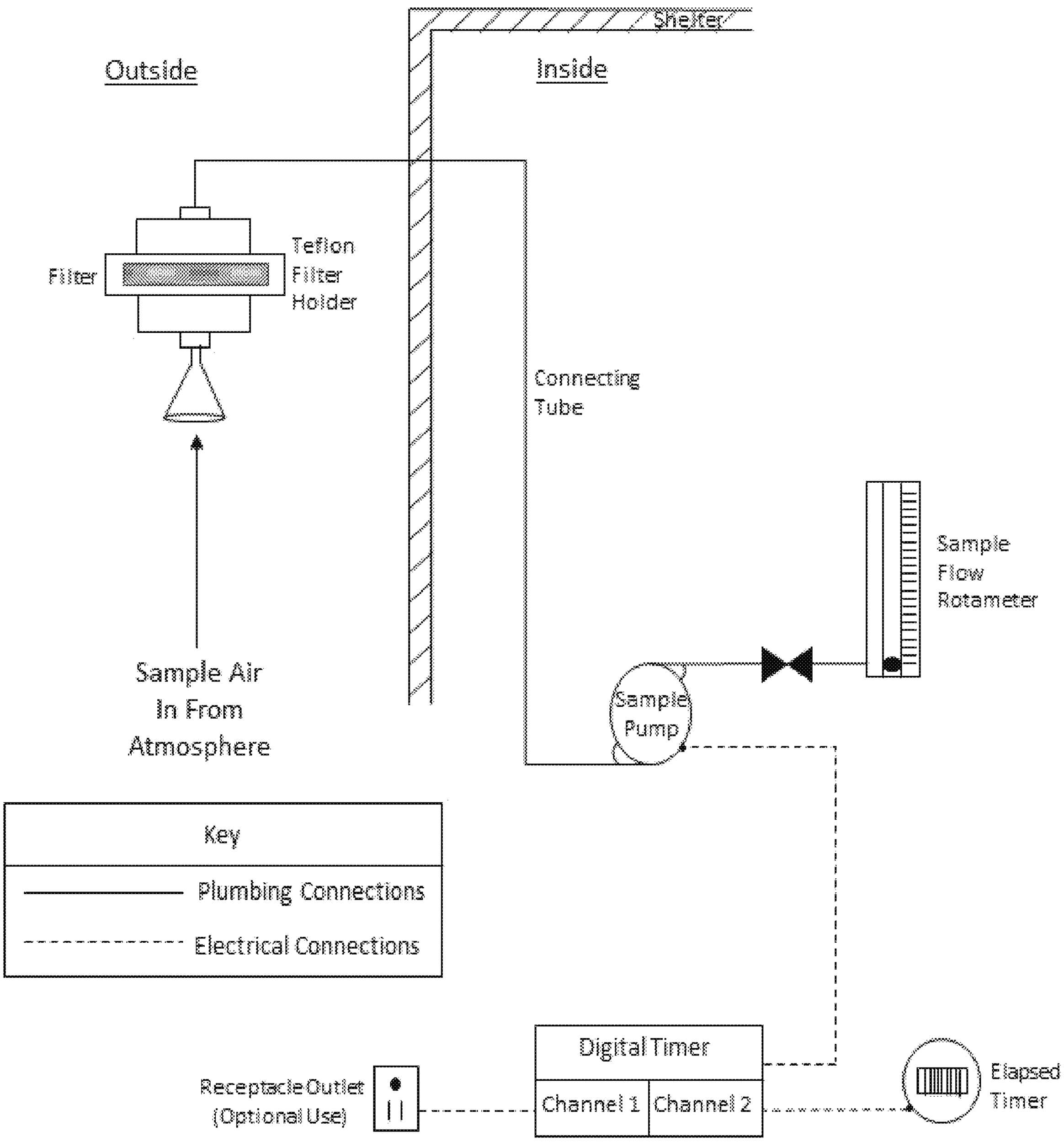

Figure 7-5. Hexavalent Chromium Sampling System Components

7.5 PAMS Sampling

PAMS sampling is performed completely by the PAMS sites in accordance with the Ozone Precursors TAD[(2)] with ERG only supplying support as requested (e.g., sampling system and training for automated gas chromatograph (GC) systems). ERG ships cleaned canisters and prepared carbonyl cartridges to the PAMS sites on the appropriate schedule to support the sampling program, and the samples are shipped to the ERG laboratory for analysis. The need for support of automated GC systems is site specific.

7.6 HAPs Sampling

HAPs sampling is performed by the sites in accordance with the methods listed in Table 3-1, with the exception of hexavalent chromium sampling (see Section 7.4). ERG provides the hexavalent chromium sampling systems and media and receives the samples from the sites for analysis.

SECTION 8
SAMPLING METHOD REQUIREMENTS

The sampling methods that are used in this program are described in this Section. Since there are four separate sampling systems and subsequently four separate analytical techniques, each of the sampling methods is different.

The SOPs for each method are reviewed annually and updated as necessary. The QA Coordinator, Program Manager and Writer/Editor will review, sign and date SOPs before distributing to the laboratories satellite file areas. The previous copies will be replaced with the revised edition. The appropriate users are notified of the updated procedure. The original, and all previously revised edits, are stored in an archive file maintained by ERG's Project Administrator.

As ERG is not responsible for actual execution of the field sampling in this program, the ERG SOPs list general sampling guidelines needed for the NMOC, UATMP, Carbonyl, and Hexavalent Chromium sampling. Table 8-1 identifies the different methods and SOP numbers for operation of each type of sampler ERG provides. Some HAPs sampling is not addressed in the NMP Support contract (Metals, PAHs, etc.), and are not discussed in this QAPP.

Table 8-1
EPA Methods and ERG SOPs for each Sampling System

Sampling System	Based on Applicable Method	ERG SOP Number
NMOC	EPA Compendium Method TO-12[3]	ERG-MOR-046
VOC	EPA Compendium Method TO-15[4]	ERG-MOR-003
Carbonyl	EPA Compendium Method TO-11A[5]	ERG-MOR-047
Hexavalent Chromium	ASTM D7614-12 Method[9]	ERG-MOR-013

SECTION 9

SAMPLE HANDLING AND CUSTODY REQUIREMENTS

Similar sample custody procedures are followed for all monitoring programs. However, program-specific differences exist because the analytical requirements for the programs vary. As these activities are conducted under one EPA contract, United Parcel Service of America (UPS) with Overnight Delivery will handle all shipping to and from the sites. Unless specified below, samples taken in the field should not require any extra special precautions for shipping.

The Shipping and Receiving Task Leader will ensure that sample media that leaves and field samples that are received in the laboratory follow all procedures listed in this QAPP and the individual SOPs. The Task Leader will also advise the Project Manager of any issues or obstacles regarding sample shipping, receipt, login and storage. The sample custodian working under the Shipping and Receiving Task Leader will ship sample media to the field and receive custody of samples, complete COC receipt information, document sample receipt, and enter COC information into LIMS to create a work order.

9.1 Canister Sample Custody

9.1.1 Canister Custody

A color-coded, three-copy canister sample COC form (Figures 9-1 and 9-2) is shipped with each 6-liter canister for the NMOC, SNMOC, UATMP, NATTS, CSATAM, or PAMS sites. If duplicate or collocated samples are to be taken, two canisters and two COC forms are sent in the shipping container(s) to the site. When a sample is collected, the site operator fills out the form per the instructions in the on-site notebook. The site operator detaches the pink copy to be retained on-site and sends the remaining copies with the canister in the shipping container to ERG's laboratory.

ERG

601 Keystone Park Drive, Suite 700, Morrisville, NC 27560

ERG Lab ID #

NMOC SAMPLE CHAIN OF CUSTODY

Lab Pre-Sampling

Site Code: ________ Canister Number: ________

City/State: ________ Lab Initial Can. Press. ("Hg): ________

AQS Code: ________ Date Can. Cleaned: ________

Collection Date: ________ Cleaning Batch # : ________

Options

NMOC (Y/N): ________

SNMOC (Y/N): ________ Duplicate Event (Y/N): ________

TOXICS (Y/N): ________ Duplicate Can # : ________

Field Setup

Operator: ________ Sys. #: ________ Rotameter Setting: ________

Setup Date: ________ Elapsed Timer Reset (Y/N): ________

Field Initial Can. Press. ("Hg): ________ Canister Valve Opened (Y/N): ________

Field Recovery

Recovery Date: ________ Sample Duration (3 or 24 hr): ________

Field Final Can. Press. (psig): ________ Elapsed Time: ________

Canister Valve Closed (Y/N): ________

Lab Recovery

Received by: ________ Date: ________ Lab Final Can. Press. (psig): ________

Status: Valid Void (Circle one)

If void, why: ________

NMOC

Analyst: ________ Database entry by: ________ Date: ________

Date: ________ Batch ID ________

NMOC Instrument: ________

Inj. 1 (AC): ________ (ppmC): ________

Inj. 2 (AC): ________ (ppmC): ________

Inj. 3 (AC): ________ (ppmC): ________

Average AC: ________

Standard Dev. (AC): ________

Average Conc. (ppmC): ________

Standard Dev. (ppmC): ________

SNMOC Option

Analyst: ________ Date: ________

Batch ID ________

Toxics Option

Analyst: ________ Date: ________

Batch ID ________

Comments: ________

White: Sample Traveler Canary: Lab Copy Pink: Field Copy

Figure 9-1. Example NMOC COC

ERG

601 Keystone Park Drive, Suite 700, Morrisville, NC 27560

ERG Lab ID #

AIR TOXICS SAMPLE CHAIN OF CUSTODY

Lab Pre-Sampling

Site Code: ____________ Canister Number: ____________

City/State: ____________ Lab Initial Can. Press. ("Hg): ____________

AQS Code: ____________ Cleaning Batch # : ____________

Collection Date: ____________ Date Can. Cleaned: ____________

Options:

SNMOC (Y/N): ____________ Duplicate Event (Y/N): ____________

TOXICS (Y/N): ____________ Duplicate Can # : ____________

METHANE (Y/N): ____________

Relinquished by: ____________ Date: ____________

Field Setup

Received by: ____________ Date: ____________

Operator: ____________ MFC Setting: ____________

System #: ____________ Elapsed Timer Reset (Y/N): ____________

Setup Date: ____________ Canister Valve Opened (Y/N): ____________

Field Initial Can. Press.: ____________ **psig psia "Hg (Circle one)**

Field Recovery

Recovery Date: ____________ Sample Duration (3 or 24 hr): ____________

Operator: ____________ Elapsed Time: ____________

Field Final Can. Press.: ____________ **psig psia "Hg (Circle one)**

Status: **VALID VOID** (Circle one) Canister Valve Closed (Y/N): ____________

Relinquished by: ____________ Date: ____________

Lab Recovery

Received by: ____________ Date: ____________

Lab Final Can. Press.: ____________ **psig "Hg (Circle one)** Converted to psia: ____________

Status: **VALID VOID** (Circle one) Gauge: 1 2 (Circle one)

If void, why: ____________

Samples stored in Air Tox Lab (Room 130)

5/2016

Comments: ____________

White: Sample Traveler Canary: Lab Copy Pink: Field Copy

Figure 9-2. Example Air Toxics COC

Upon receipt, the sample canister vacuum/pressure is measured and compared against the field documented vacuum/pressure to ensure the canister remained airtight during transport. If the receiving vacuum differs from the field vacuum more than 3"Hg, the program manager is notified, and sample canister may be voided. Because there are potential differences in barometric pressures and temperatures between the sampling site and the receiving laboratory (such as those sites at high altitudes), and different accuracies for different types of pressure gauges, there can be a consistent difference in final field pressure and lab receipt pressure for canister samples. This difference and other parameters are considered to determine the validity of the canister samples. These are monitored daily and the pressures are logged into an Excel spreadsheet. This allows the laboratory the ability to determine if the difference is due to gauges or if the canister leaked en route. A sample of the spreadsheet is presented in Table 9-1.

Table 9-1

Example of Canister Pressure Check Spreadsheet

Date Received	Site	Field Pressure Reading	Lab Pressure Reading	Difference
8/30/16	NBIL	2 "Hg	6 "Hg	4 "Hg
9/7/16	NBIL	1 "Hg	4 "Hg	3 "Hg
9/14/16	NBIL	3 "Hg	7 "Hg	4"Hg
9/16/16	NBIL	4 "Hg	7 "Hg	3 "Hg
8/30/16	BLKY	5 "Hg	5 "Hg	0 "Hg
9/7/16	BLKY	5 "Hg	3.5 "Hg	1.5 "Hg
9/13/16	BLKY	5 "Hg	5 "Hg	0 "Hg
9/16/16	BLKY	5 "Hg	4 "Hg	1 "Hg

The canister should be cleaned no more than 30 days before sampling. If the canister is older than 30 days, a note will be made in LIMS and a flag will be added to the sample results in AQS. More detailed sample receipt procedures and sample acceptance policies are presented in the *SOP for Sample Receipt at the ERG Chemistry Laboratory*, ERG-MOR-045 in Appendix C. The sample specific information from the COC is then entered into LIMS (example login page is shown in Figure 9-3) following the *SOP for Sample Login to the Laboratory Information Management System*, ERG-MOR-079 found in Appendix C. The sample is given a unique LIMS identification (ID) number and tagged (see Figure 9-4), noting the site location and the sample collection date.

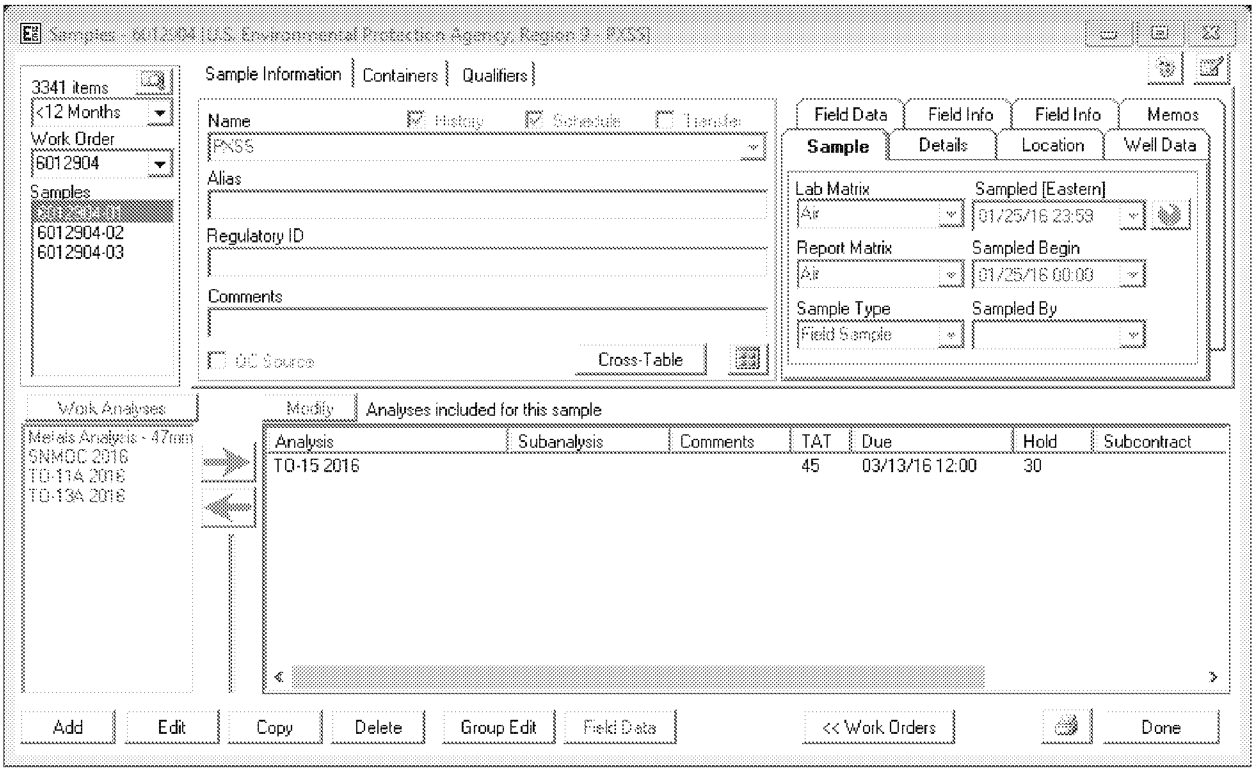

Figure 9-3. Example ERG LIMS Login Page

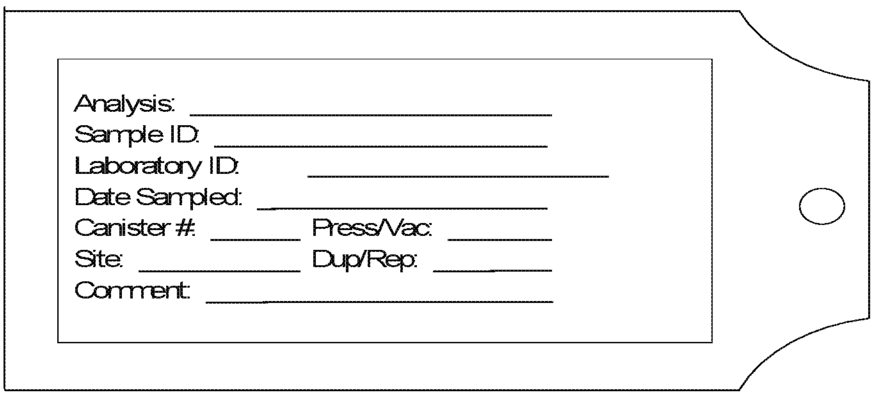

Figure 9-4. Canister Tag

The LIMS ID number is recorded on the canister tag and on all ERG copies of the COC. The remaining copies of the canister sample COC are separated. The white copy is scanned (the

PDF is stored in the LIMS system) and is kept with the canister sample until analysis is complete. After sample analysis, the white copy goes into the data package with the sample data. The yellow copy is stored chronologically in a designated file cabinet for one year. The file cabinet is in Room 102 in the Laboratory building.

9.1.2 Canister Analytical Routing Schedule

Each canister has a unique canister identification number inscribed on the canister. This number is used during can cleaning, field collection, laboratory receipt, and laboratory sample analysis and is included on the individual Toxics/SNMOC COCs and entered into the LIMS.

The canister sample analysis hold time is 30 days from the sampling date. The samples are sent to the ERG Air Toxics Laboratory for VOC and SNMOC/PAMS GC/Flame Ionization Detector/Mass Spectrometer (FID/MS) analysis. The canister sample is analyzed and kept in the laboratory until after the analyst reviews the relevant analytical data.

9.1.3 Canister Cleanup

All canisters are cleaned prior to reuse following SOP ERG-MOR-105 (*SOP for Sample Canister Cleaning using Wasson TO-Clean Automated System*) as shown in Appendix C. The canisters are cleaned using the procedure described in Section 10.1.1. The unheated system (following SOP ERG-MOR-062, *SOP for Sample Canister Cleaning*) is maintained as a backup, if needed, and is described in Section 10.1.2. The canisters are cleaned to <3x MDL or 0.2 parts per billion by volume (ppbV), whichever is lower, and 20 parts per billion as Carbon (ppbC) for Total SNMOC. If the canister fails the Blank criteria, it is returned to the cleaning system bank with the other canisters that were cleaned along with it and all canisters are put through an additional Vacuum and Pressure cycle. The same canister is analyzed again. All canisters, whether used for NMOC, SNMOC, UATMP, NATTS, CSATAM, or PAMS, are cleaned by the same procedure and are entered into the canister cleanup log, shown in Figure 9-5 for the heated systems and in Figure 9-6 for the unheated systems.

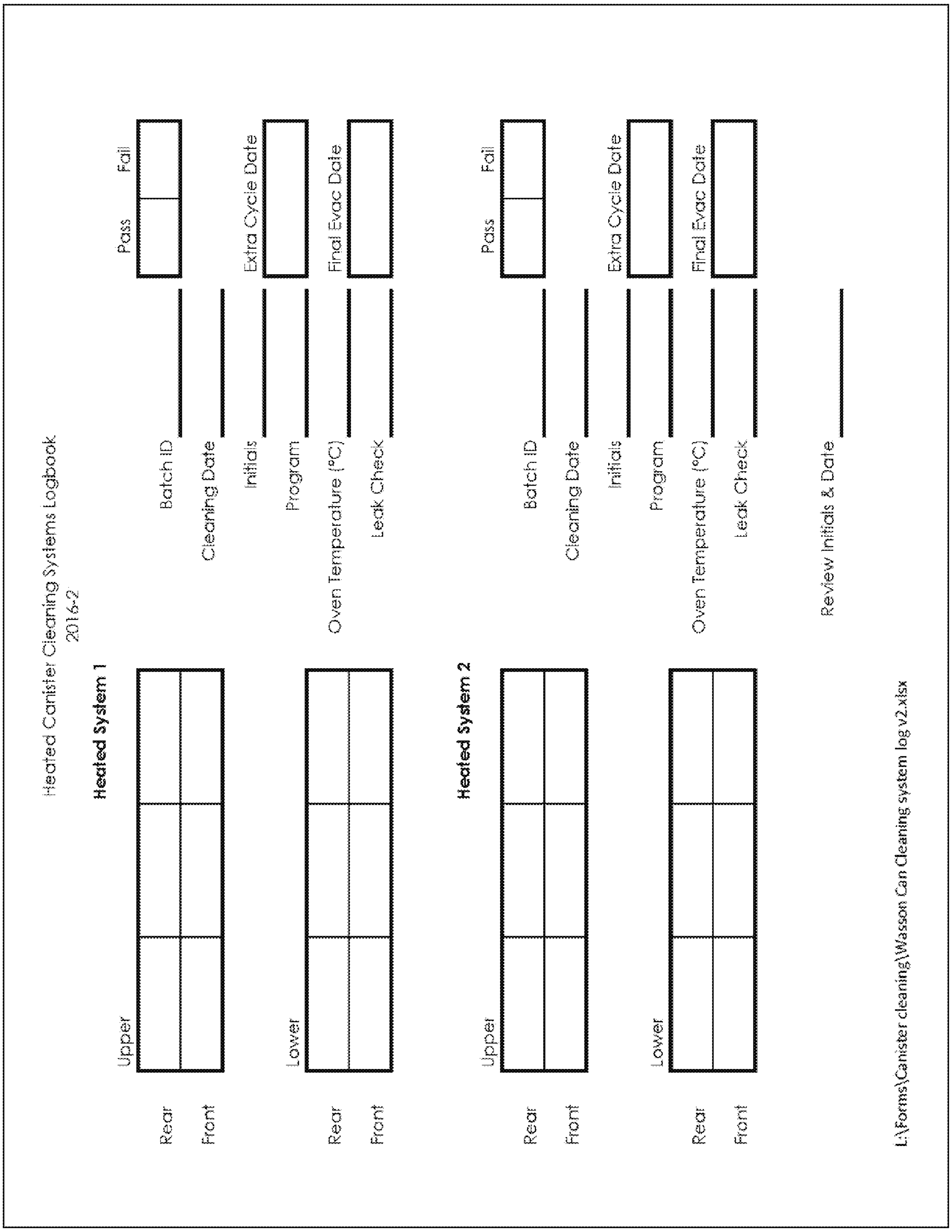

Figure 9-5. Canister Cleanup Log for the ERG Heated Cleaning System

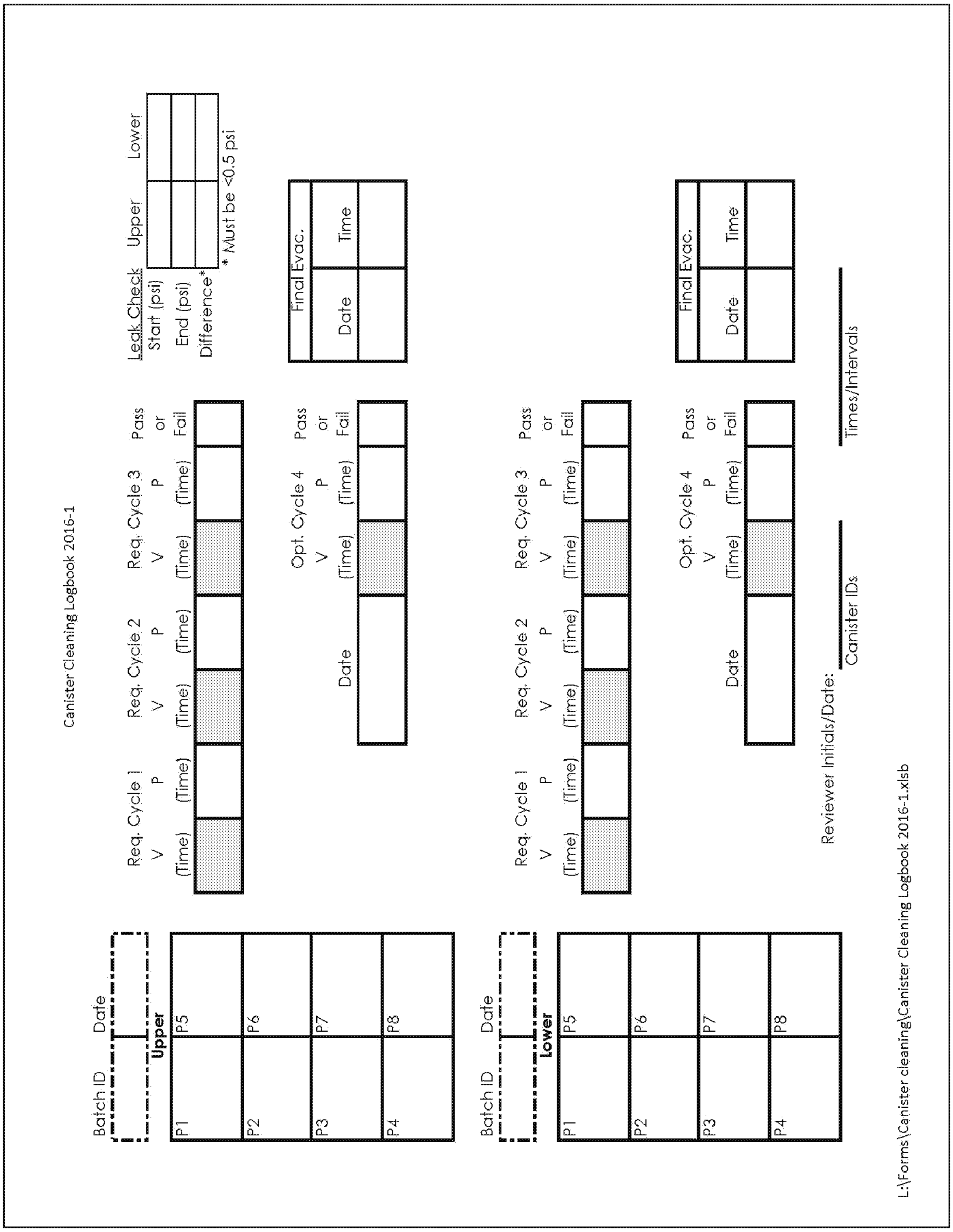

Figure 9-6. Canister Cleanup Log for the ERG Unheated Cleanup System

9.2 Carbonyl Sample Custody

Figure 9-7 shows the color-coded, three-copy COC form used for all carbonyl sampling documentation. A COC is shipped to the site with the carbonyl cartridges. After sampling, the COC form is completed by the site operator and the pink copy is retained for site records. The carbonyl sample cartridges and remaining COC copies are shipped to ERG's analytical laboratory.

When samples are received, they are logged into the LIMS database and given a unique LIMS ID number following the *SOP for Sample Login to the Laboratory Information Management System,* SOP ERG-MOR-079, found in Appendix C. The remaining copies of the COC are separated. The white copy of the COC is scanned (the PDF is stored in the LIMS system) and is labeled with the LIMS ID number, site code, sampling date, individual sample designations, and date of receipt and initials of receiving personnel and put into a bag. The sample bag is stored in a refrigerator designated for carbonyl samples only. The yellow copy is stored chronologically in a designated file cabinet for one year. The file cabinet is in Room 102 in the Laboratory building. More detailed sample receipt procedures and sample acceptance policies are presented in the *SOP for Sample Receipt at the ERG Chemistry Laboratory,* ERG-MOR-045.

9.2.1 Carbonyl Analytical Routing Schedule

The carbonyl cartridge samples are extracted within 14 days of the sampling day and analyzed within 30 days after extraction. The extracts are kept in the designated extract refrigerator until after the analyst and the Task Leader reviews all the relevant analytical data.

ERG Lab ID #

601 Keystone Park Drive, Suite 700, Morrisville, NC 27560

CARBONYL COMPOUNDS CHAIN OF CUSTODY

Lab Pre-Samp.

Site Code: ________ Collection Date: ________
City/State: ________ Cartridge Lot #: ________
AQS Code: ________ Duplicate Event (Y/N): ________
Relinquished by: ________ Date: ________

Field Setup

Received by: ________ Date: ________
Set-Up Date: ________ Operator: ________ Sys. #: ________
Pre-Sampling Rotameter Reading (cc/min): ________ Elapsed Timer Reset (Y/N): ________

Field Recovery

Recovery Date: ________ Sample Duration (3 or 24 hr): ________
Operator: ________ Elapsed Time: ________
Post Sampling Rotameter Reading (cc/min): ________ Status: **VALID** **VOID** (Circle one)
Cartridges Capped (Y/N): ________
Relinquished by: ________ Date: ________

Lab Recovery

Received by: ________ Date: ________
Status: **VALID** **VOID** (Circle one) Uncorrected Temperature: ________
If void, why: ________ Corrected Temperature: ________
Sample Volume (total Liters): ________ IR Gun: 1 2 (Circle one)
Samples stored in Refrigerator # 11

PAMS

Sample Date	Sample Time	Sample Duration	Sample Volume	Cartridge Lot #	Sample ID	Lab ID

Comments: ________

White: Sample Traveler Canary: Lab Copy Pink: Field Copy

Figure 9-7. Example Carbonyl Compounds COC

9.3 HAPs Sample Custody

Samples collected on prepared sample media (i.e., XAD-$^{2®}$, Polyurethane Foam (PUF), hexavalent chromium filters, etc.) use supplied three-copy COC forms to document sample collection. Field testing personnel will record applicable collection data (such as time, date, location, meteorological parameters) on the appropriate COC forms (Figures 9-8, 9-9 and 9-10) and keep the pink copies for site records. The COCs are then shipped to ERG with the prepared sample media.

Because the sites supply the filters used for metal analysis, COC forms are normally supplied by the State, Local or Tribal agency for these samples. If needed, however, COC forms can be supplied by ERG electronically inputting multiple filters for metal analysis (Figure 9-11). Samples are received at ERG's laboratory as presented in the *SOP for Sample Receipt at ERG Chemistry Laboratory*, ERG-MOR-045.

All HAPs samples received at the ERG laboratory will be logged into the LIMS as described in the *SOP for Sample Login to the Laboratory Information Management System,* ERG-MOR-079.

9.4 Invalid Samples

The sample COC form may indicate that the sample sent from a site is invalid. The sample can be determined invalid at the site or in the laboratory. SOP ERG-MOR-045 describes the sample receiving procedure and sample acceptance. Individual sites will be contacted if there are any questions about the samples upon receipt. When a sample is designated as invalid, the assigned LIMS ID number is notated as a void and is invalidated on the individual respective COC form. Another sample media will be sent to the site with the COC designated to make up on non-standard sampling days. If the site has repeated invalid samples, normally three voids in a row, the ERG site coordinator Task Leader will work with the site personnel to diagnose and correct the problem. The sites will also be notified in the monthly analytical reports of any invalid samples.

ERG Lab ID # ______

501 Keystone Park Drive, Suite 700, Morrisville, NC 27560

SVOC SAMPLE CHAIN OF CUSTODY

Lab Pre-Sampling

Site Code: ______ Container #: ______
City/State: ______ Collection Date: ______
AQS Code: ______ Collocated Event (Y/N): ______
SUR ID: ______
Cartridge Certification Date: ______ XAD Lot: ______
PUF Lot: ______
Relinquished by: ______ Date: ______ Filter Lot: ______

Field Setup

Received by: ______ Date: ______
Site Operator: ______ System #: ______
Set-Up Date: ______ Elapsed Timer Reset (Y/N): ______

Field Recovery

Recovery Date: ______

Collection System Information:

	Elapsed Time	Temp (°C)	Barometric ("Hg)	Magnehelic ("H_2O)	Flowrate (std. m^3/min)
Start					
End					
Average					

Total Collection Time (Minutes) ______ Total Collection Volume (std. m^3) ______
Status: **Valid** **Void** (Circle one) Site Operator: ______
Relinquished by: ______ Date: ______

Lab Recovery

Received by: ______ Date: ______ Container #: ______
Status: **Valid** **Void** (Circle one) Uncorrected Temperature: ______
If void, why: ______ Corrected Temperature: ______
Thermometer: **IR1** **IR2**
(Circle one)

Samples stored in Refrigerator # 7

Comments: ______

White: Sample Traveler Canary: Lab Copy Pink: Field Copy

Figure 9-8. Example SVOC Sample COC

ERG

601 Keystone Park Drive, Suite 700, Morrisville, NC 27560

ERG Lab ID # ____

AMBIENT HEXAVALENT CHROMIUM CHAIN OF CUSTODY FORM

Lab Pre-Sampling

Site Code: ____ Collection Date: ____

City/State: ____ Primary Event (Y/N): ____

AQS Code: ____ Collocated Event (Y/N): ____

Relinquished by: ____ Date: ____

Field Setup

Received by: ____ Date: ____

Site Operator: ____ System #: ____

Set-Up Date: ____ Elapsed Timer Reset (Y/N): ____

Collection Date: ____

Batch I.D. No.: ____

Initial Rotameter Setting (C.O. B.): ____ (After 5 minutes warm-up)

Programmed Start Time: ____ Programmed End Time: ____

Field Recovery

Recovery Date: ____ Recovery Time: ____

Site Operator: ____

Final Rotameter Reading (C.O.B.): ____ (After 5 minutes warm-up)

Elapsed Time: ____ Status: Valid Void (Circle one)

Relinquished by: ____ Date: ____

Lab Recovery

Received by: ____ Date: ____ Container #: ____

Status: Valid Void (Circle one) Uncorrected Temperature: ____

If void, why: ____ Corrected Temperature: ____

Collection Time (Minutes): ____ IR Gun: 1 2 (Circle one)

Avg. Flowrate (L/min): ____

Total Volume of Air Sampled (m^3): ____

Samples stored in Freezer # 11

Comments: ____

White: Sample Traveler Canary: Lab Copy Pink: Field Copy

Figure 9-9. Example Ambient Hexavalent Chromium COC

ERG

ERG Lab ID #

601 Keystone Park Drive, Suite 700, Morrisville, NC 27560

PM$_{10}$ / TSP METALS CHAIN OF CUSTODY

Lab Pre-Samp.

Site Code: ____________ Collection Date: ____________

City/State: ____________ Duplicate Event (Y/N): ____________

AQS Code: ____________

Relinquished by: ____________ Date: ____________

Field Setup

Received by: ____________ Date: ____________

Set-Up Date: ____________ Operator: ____________

Field Recovery

Recovery Date: ____________ Sample Duration (i.e. 24 hr): ____________

Status: Valid Void (Circle one)

Relinquished by: ____________ Date: ____________

Lab Recovery

Received by: ____________ Date: ____________

Status: Valid Void (Circle one)

If void, why: ____________

Samples stored in ICP-MS Lab (Room # 128)

PM$_{10}$ / TSP METALS

Sample Date	Start Time	End Time	Total Time	System #	Total Vol (m^3)	Lab ID
	Start MFC	End MFC	Avg Flow (L/min)	Filter #		
Sample Date	Start Time	End Time	Total Time	System #	Total Vol (m^3)	Lab ID
	Start MFC	End MFC	Avg Flow (L/min)	Filter #		
Sample Date	Start Time	End Time	Total Time	System #	Total Vol (m^3)	Lab ID
	Start MFC	End MFC	Avg Flow (L/min)	Filter #		

8-2017

Comments: ____________

White: Sample Traveler Canary: Lab Copy Pink: Field Copy

Figure 9-10. Example Metals COC

ERG

Chain of Custody Record

601 Keystone Park Drive, Suite 700, Morrisville, NC 27560

Page __________ of __________

PROJECT			NO. OF CONTAINERS	ANALYSES					STORAGE LOCATION	
SITE										
COLLECTED BY (Signature)										
FIELD SAMPLE I.D.	SAMPLE MATRIX	DATE/TIME							REMARKS	ERG LIMS ID (For lab use only)

REMARKS:

RECEIVED BY:	DATE	TIME	RELINQUISHED BY:	DATE	TIME	RECEIVED BY:	DATE	TIME	RELINQUISHED BY:	DATE	TIME

LAB USE ONLY

RECEIVED FOR LABORATORY BY:	DATE	TIME	AIRBILL NO.	OPENED BY:	DATE	TIME	TEMP °C	SEAL #	CONDITION

REMARKS:

8-2017

White: Sample Traveler   Canary: Lab Copy   Pink: Field Copy

Figure 9-11. ERG Blank COC Record

9.5 Analytical Data

After analysis, the laboratory will provide narratives describing any anomalies and modifications to analytical procedures, data and sample handling records, and laboratory notes for inclusion in the final report. All laboratory electronic records will be stored for archive on Digital Versatile Disk (DVD), or shared network drive. DVDs are stored in Room 102 in the Laboratory building and the shared network has limited access. Raw data will be stored on the shared network for at least 5 years after the end of the closed contract.

All records generated by measurement activities are signed or initialed by the person performing the work and reviewed by an appropriate Task Leader. Measurement results become part of a project report, of which 10 percent is requested by the QA Coordinator (or a reviewer designated by the QA Coordinator) for review.

9.6 Sampling Monitoring Data

All COC forms from the monitoring sites will be stored with the analytical results. The forms are also scanned and stored in the LIMS as described in the *SOP for Sample Login to the Laboratory Information Management System,* SOP ERG-MOR-079. The COC forms will be reviewed by the sample custodian(s), Task Leaders and Program Manager. The laboratory will contact the individual site if necessary information is not completed on the COC forms. The original field data will remain in ERG custody and will eventually be stored on file with the final report until 5 years after the end of the closed contract.

SECTION 10
ANALYTICAL METHODS REQUIREMENTS

Analytical procedures are program-specific because the instrumentation and the target compounds of the four programs differ. The primary analytical instrument is GC/FID/MS for SNMOC, VOCs and PAMS hydrocarbons; High Performance Liquid Chromatography (HPLC) for carbonyls; GC/MS for Semivolatiles (SVOC); Inductively Coupled Plasma/Mass Spectrometer (ICP-MS) for Metals; and Ion Chromatography (IC) for Hexavalent Chromium. All samples taken for SNMOC, VOCs, or PAMS hydrocarbons can be evaluated by GC/FID/MS because the instrumentation is collecting all of the data at the same time. Corrective action for analytical system failures realized at time of analyses is initiated by the Analyst and supported by the Task Leader for that method. All analytical method SOPs are provided in Appendix C. The methods used for NMOC and other individual HAPs analysis not currently discussed will be added to this QAPP when the individual States request the analyses. Samples will not be analyzed until ERG receives approval from EPA.

The SOPs for each method are reviewed annually and updated as necessary. The QA Coordinator, Program Manager and Writer/Editor will review, sign and date SOPs before distributing to the laboratories satellite file areas. The previous copies will be replaced with the revised edition. The original, and all previously revised edits, are stored in a historical file maintained by ERG's Project Administrator.

10.1 Canister Cleanup System

The canisters are cleaned using a Wasson TO-Clean Model TO 0108 heated canister cleaning system and is explained in Section 10.1.1. The unheated system is used as backup and is described in Section 10.1.2. A bulk liquid N_2 dewar is located external to the ERG laboratory facility. This dewar continuously produces a volume of ultrapure gaseous N_2 in its headspace area (~100 psig) that is more than adequate to accommodate all in-lab gaseous N_2 applications. Ultrapure gaseous N_2 is extracted from the dewar headspace and delivered to the cleaning systems. Transport of the gas is accomplished through a 3/8" outer diameter (OD) pre-cleaned stainless-steel tubing.

10.1.1 Heated Canister Cleaning System

The TO-Clean heated cleaning systems are commercially available systems manufactured by Wasson-ECE (Figure 10-1). These systems can clean up to twelve canisters per system at a selected temperature from ambient to 100°C. Each system consists of an oven that holds the canisters, an Edwards RV8 vacuum pump, a stainless-steel humidification chamber for the dilution gas, and a control unit. The procedure for cleaning canisters is the *SOP for Sample Canister Cleaning using the Wasson-ECE,* ERG-MOR-105 in Appendix C.

The cleaning system oven has enough capacity to clean up to 12 canisters at a time. Two racks hold up to six canisters each. Canisters are connected to a 12-port, two-level manifold with compression fittings and flexible stainless-steel tubing. Ultra-pure N_2 is the dilution gas and is applied to the manifold via an electrically actuated valve. Vacuum is applied to the manifold through a pneumatically-actuated vacuum valve. The oven is heated to 40°C during the cleaning cycles.

The control unit controls the pressure, vacuum, and vent valves and houses the front panel control unit and oven temperature controller. The touchscreen front panel control stores and executes the cleaning programs, provides manual valve control and leak check diagnostics, and displays vacuum, pressure, and program time information. The oven temperature controller is separate from the front panel control within the control unit and regulates the oven temperature to a preset value.

The Edwards RV8 vacuum pump is separated from the system by a cryogenic trap. This trap removes contaminants and water vapor from the canisters before reaching the pump, and it prevents the sample canisters from being contaminated by back-diffusion of hydrocarbons from the vacuum pump into the cleanup system. The humidifier system is a modified SUMMA®-treated 6-liter canister partially filled with HPLC-grade water. The ultra-pure N_2 dilution gas is

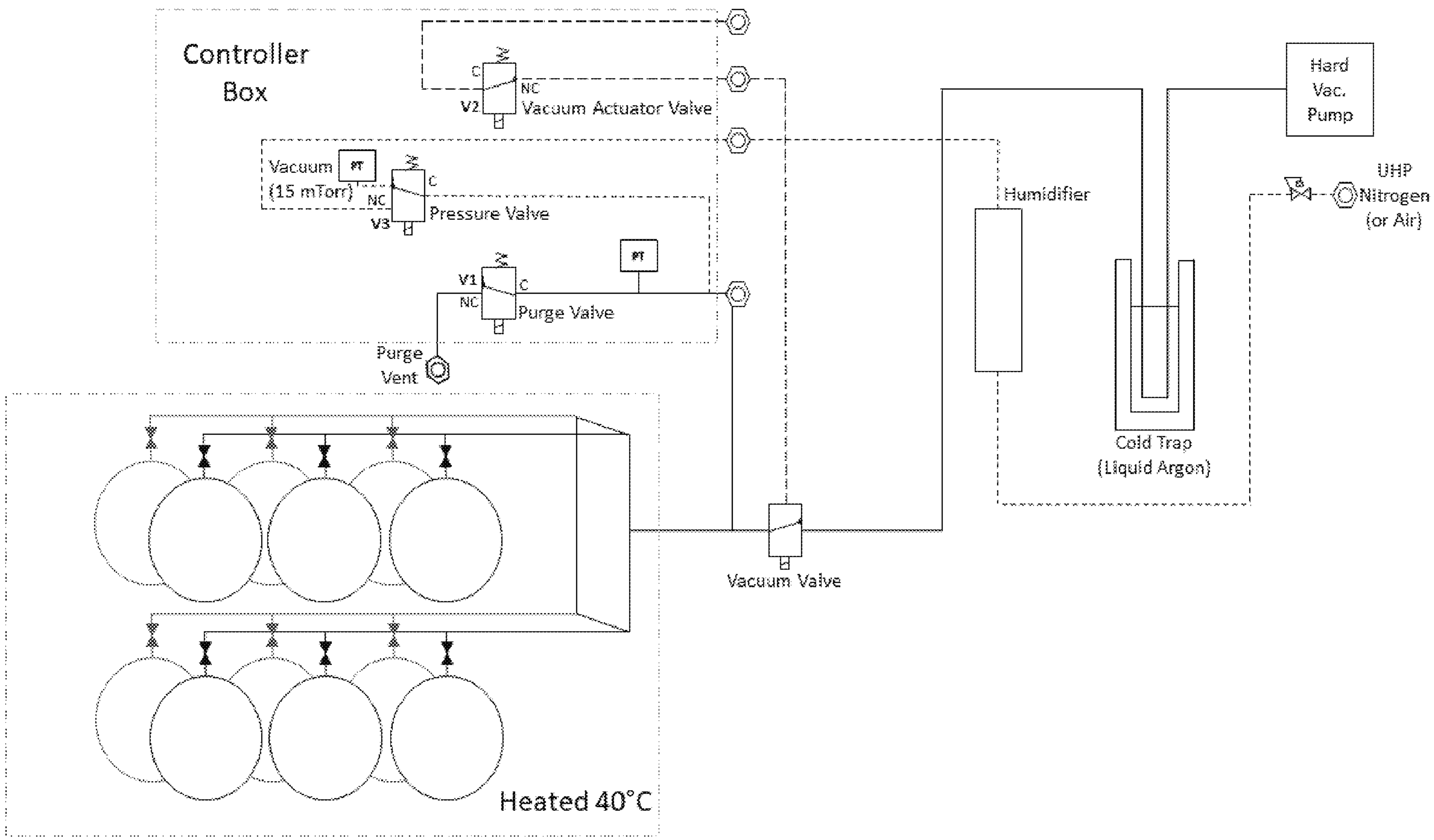

Figure 10-1. Heated Canister Cleanup System Schematic

bubbled through the water prior to entering the manifold, achieving an estimated relative humidity of 75 percent.

After sample analyses and data review are completed, 12 canisters are connected to the manifold in the oven. The bellows valve on each canister is opened. The vacuum pump is started and one of the vacuum routing valves is opened, drawing a vacuum on the canisters connected to the corresponding manifold. The canisters are evacuated to a vacuum reading of 400 millitorr and held for 45 minutes. The vacuum valve is then closed and the ultrapure gaseous N_2 that has been humidified is introduced into the evacuated canisters at a rate of 5.0 liters per minute until the pressure in the canisters reach approximately 20 psig. This evacuation and pressurization of the canisters constitutes one Cleanup Cycle.

The Cleanup Cycle is repeated twice more to facilitate a complete canister cleanup procedure. Following the third pressurization, the canister bellows valves are closed and one canister (out of the 12 cleaned) is selected for cleanliness verification analysis. The cleanliness of the canister is qualified by GC/MS and FID analysis. The pass/fail results of the analyses are documented on a shared network so that the pass/fail rate can be monitored. The cleanliness criterion for each bank of 12 canisters is < 3x MDL or 0.2 ppbV for each individual VOC, whichever is lower, and 20 ppbC for Total SNMOC. If the canister does not pass the cleanliness criteria, the canister is reconnected to the cleanup manifold with the other 11 canisters it was cleaned with and another cleaning cycle is performed, and the same canister is analyzed again. Upon meeting these criteria, the canister is reconnected to the cleanup manifold with the other 11 canisters constituting the original bank of 12. All 12 canister bellows valves are opened, and the canisters are evacuated to a vacuum reading of 50 millitorr. The bellow valves are closed, and canisters are ready to be packaged and shipped to each network site.

10.1.2 <u>Unheated Canister Cleaning System</u>

A canister cleanup system (Figure 10-2) has been developed and is used to prepare sample canisters for use in collecting representative whole air samples (*SOP for Sample Canister*

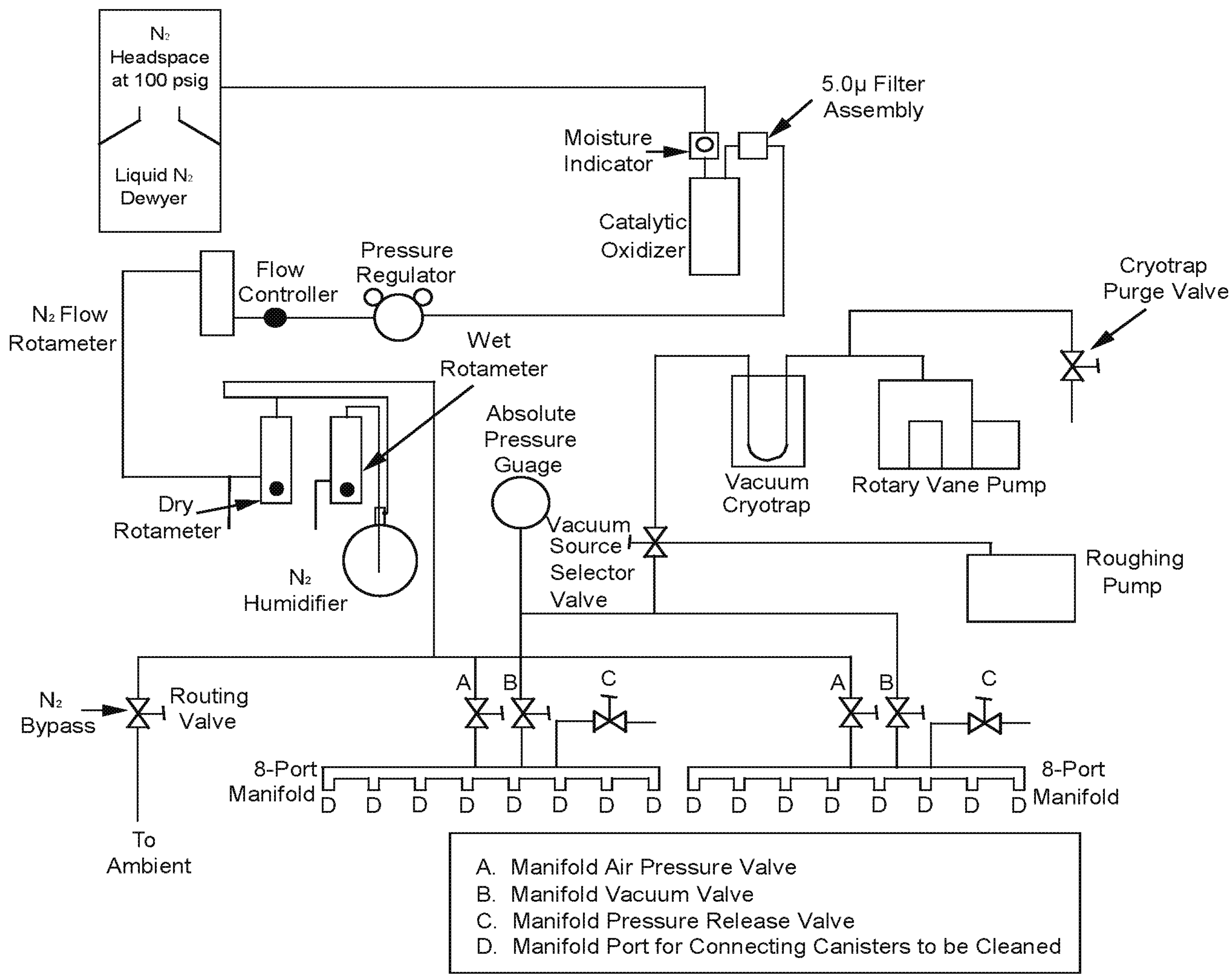

Figure 10-2. Unheated Canister Cleanup System Schematic

Cleaning, ERG-MOR-062 in Appendix C). This cleaning system is used as a backup to the heated canister cleaning system explained in Section 10.1.1.

A single-stage regulator controls the final N_2 pressure in the canisters and a metering valve is used to control the flow rate at which the canisters are filled during a cleanup cycle. The flow direction is controlled by a separate flow meter, installed in the N_2 gas line. A shutoff valve exists between the N_2 gas line and the humidifier system (which is a modified SUMMA®-treated 6-liter canister partially filled with HPLC-grade water). One rotameter and flow-control valve direct the gaseous N_2 into the humidifier where it is bubbled through the HPLC-grade water. A second flow-control valve and flow meter allow gaseous N_2 to bypass the humidifier system, if desired. By setting the flow-control valves separately, the downstream relative humidity can be regulated. Approximately 75 percent relative humidity is used for canister cleaning. This is accomplished by routing 100 percent of the gaseous N_2 flow through the humidifier. Another shutoff valve is located between the humidifier and each 8-port manifold where the canisters are connected for cleanup.

The vacuum system consists of a Precision Model DD-310 vacuum pump, a cryogenic trap, a vacuum and pressure gauge, and a manifold vacuum valve connected as shown in Figure 10-1. The cryogenic trap prevents the sample canisters from being contaminated by back-diffusion of hydrocarbons from the vacuum pump into the cleanup system. The manifold vacuum valves enable isolation of the vacuum pump from the system without shutting off the vacuum pump.

After sample analyses and data review are completed, a bank of eight canisters is connected to each manifold as shown in Figure 10-1. The canister bellows valve on each canister is opened. The vacuum pump is started and one of the vacuum routing valves is opened, drawing a vacuum on the canisters connected to the corresponding manifold. The bank of eight canisters is evacuated to a vacuum reading of 29.5" Hg (as indicated by the pressure gauge), and held for 30 minutes. The vacuum routing valves are then closed and the ultrapure gaseous N_2 that has been humidified is introduced into the evacuated canisters at a rate of 4.0 liters per minute until the pressure in the canisters reach approximately 20 psig. This "Evacuation and Pressurization" of the canisters constitutes one Cleanup Cycle.

The Cleanup Cycle is repeated twice more to facilitate a complete canister cleanup procedure. Following the third pressurization, the canister bellows valves are closed and one canister (out of the eight cleaned) is selected for cleanliness verification analysis. The cleanliness of the canister is qualified by GC/MS and FID analysis. The pass/fail results of the analyses are documented on a shared network so that the pass/fail rate can be monitored. The cleanliness criterion for each bank of eight canisters is < 3x MDL or 0.2 ppbV for each individual VOC, whichever is lower, and 20 ppbC for Total SNMOC. If the canister does not pass the cleanliness criteria, the canister is reconnected to the cleanup manifold with the other seven canisters it was cleaned with and another cleaning cycle is performed, and the same canister is analyzed again. Upon meeting these criteria, the canister is reconnected to the cleanup manifold with the other seven canisters constituting the original bank of eight. All eight canister bellows valves are opened and the canisters are evacuated to a vacuum reading of approximately 29.5" Hg for a fourth time. The bellow valves are closed, and the canisters are ready to be packaged and shipped to each network site.

10.2 VOC and Concurrent Analytical System

The VOC GC/FID/MS analyses are performed on a 250-milliliter (mL) sample from the canister with an Agilent 6890 GC/FID and an Agilent 5975 MS with Selected Ion Monitoring (SIM) using a 60 m by 0.32-millimeter (mm) Inner Diameter and a 1-micrometer (μm) film thickness Restek R_{xi}-1_{ms} capillary column followed by a Y-union connector that splits the mobile phase between the MS and the FID. Table 10-1 shows the GC/FID/MS operating conditions. Figure 10-3 shows the GC/FID/MS system arrangement. Canister samples must be analyzed within 30 days from sample collection. The analytical *SOP for the Concurrent GC/FID/MS Analysis of Canister Air Toxic Samples using EPA Compendium Method TO-15 and EPA Ozone Precursor Method* (ERG-MOR-005) is presented in Appendix C.

Table 10-1

VOC GC/FID/MS Operating Conditions

Parameter	Operating Value
Sample Volume	250 mL
Restek R_{xi}-1_{ms} Capillary Column: Length: Inside diameter: Film thickness: Oven temperature:	 60 m 0.32 mm 1 µm -50°C for 5 minutes, 15°C/min to 0°C then 5°C/min to 150°C, then 25°C/min to 220°C for 1 minute then 25°C/min to 150°C for 4 minutes
Temperatures: FID: Injector Oven Temperature: MS Quad Temperature: MS Source Temperature:	 300°C 220°C 200°C 280°C (350°C 5975)
Gas Flow Rates: Column Carrier Gas (Helium (He)): FID Make-up (He): FID (Hydrogen (H_2)): FID (Air):	 2 mL/min 30 mL/min 30 mL/min 300 mL/min
Entech Sample Interface Conditions: Module 1 - Glass Bead/Tenax® Trap Initial Temperature: Module 2 - Tenax® Trap Initial Temperature: Module 3 - Cryofocuser Temperature:	 -150°C -50°C -196°C

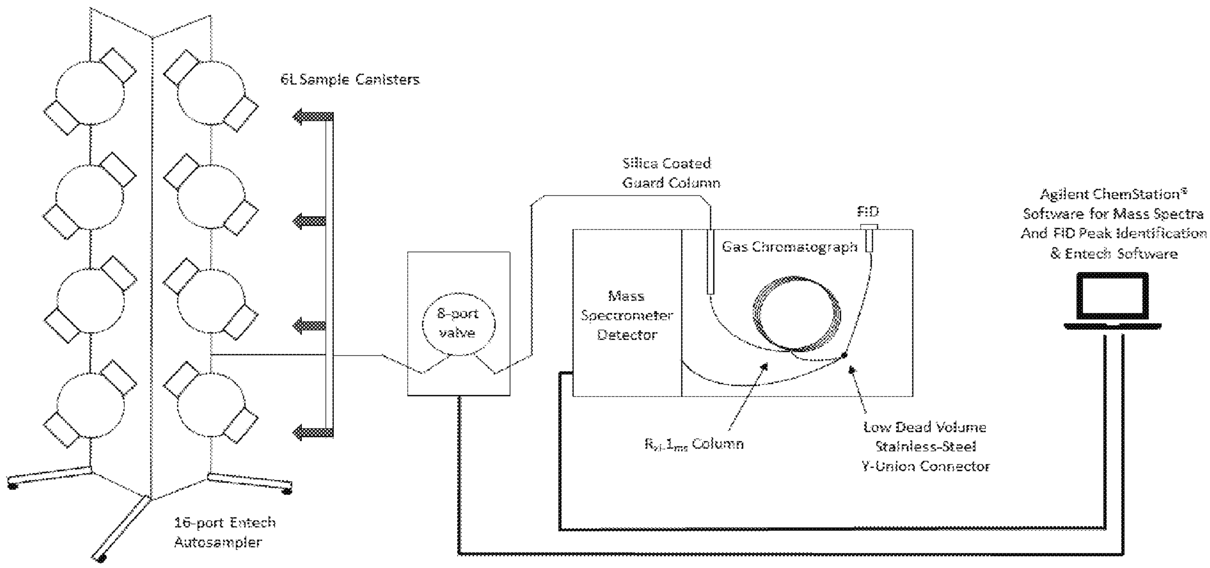

Figure 10-3. VOC GC/MS/FID System

10.3 Carbonyl Analytical System

Carbonyl samples are stored in the refrigerator after they are received from the field prior to analysis. The carbonyl cartridge samples are extracted within 14 days of the sampling day and analyzed within 30 days after extractions. Sample preparation is performed by removing the DNPH sampling cartridge from its shipping container and attaching it to the end of a 5 mL Micro-Mate® glass syringe. Five mL of acetonitrile are added to the syringe and allowed to drain through the cartridge into a 5 mL Class A volumetric flask and diluted to the 5 mL mark with acetonitrile. This solution is then transferred to a 2 mL autosampler vial fitted with a Teflon-lined, self-sealing septum and a 4 mL vial with a Teflon-lined cap and both vials are stored in a refrigerator at 4°C until analysis.

The analytical separation of carbonyls is performed using a Waters HPLC configured with a reverse-phase 250 mm by 4.6 mm C-18 silica analytical column with a 5-micron particle size. A typical HPLC system is shown in Figure 10-4. ERG's system uses an Agilent HPLC chromatographic data software system. Typically, 15-microliters (µL) samples are injected with an automatic sample injector. A mobile phase gradient of water, acetonitrile, and methanol is used to perform the analytical separation at a flow rate of 1.0 mL/minute. A multiwavelength Ultraviolet (UV) detector is operated at 360 nanometer (nm). The complete *SOP for Preparing, Extracting, and Analyzing DNPH Carbonyl Cartridges by Method TO-11A* (ERG-MOR-024) is presented in Appendix C. Sample and waste disposal procedures are outlined in ERG-MOR-033, the *SOP for Hazardous Waste*.

10.4 Polycyclic Aromatic Hydrocarbons Analytical Systems

Sampling modules containing PUF/XAD-2®, petri dishes containing glass microfiber filters, and COC forms and all associated documentation will be shipped to the ERG laboratory from the field. Each filter should be folded in quarters, placed inside the cartridge (with the XAD/PUF) and capped before shipment. Upon receipt at the laboratory, samples will be logged into the LIMS system and stored in the refrigerator. Sample preparation and analysis procedures are based on EPA Compendium Method TO-13A[(10)] and ASTM D6209-13[(12)] method. The hold time is 14 days after sampling for extraction and 40 days after extraction for analysis.

Sample extracts will be analyzed for PAHs using GC/MS in SIM. The MS will be tuned and mass-calibrated as required using perfluorotributylamine (FC-43), per the analytical procedures presented in the *SOP for analysis of Semivolatile Organic Compounds (Polynuclear Aromatic Hydrocarbons) Using EPA Compendium Method TO-13A and ASTM D6209* (ERG-MOR-049) (see Appendix C). Sample and waste disposal procedures are outlined in ERG-MOR-033, the *SOP for Hazardous Waste*.

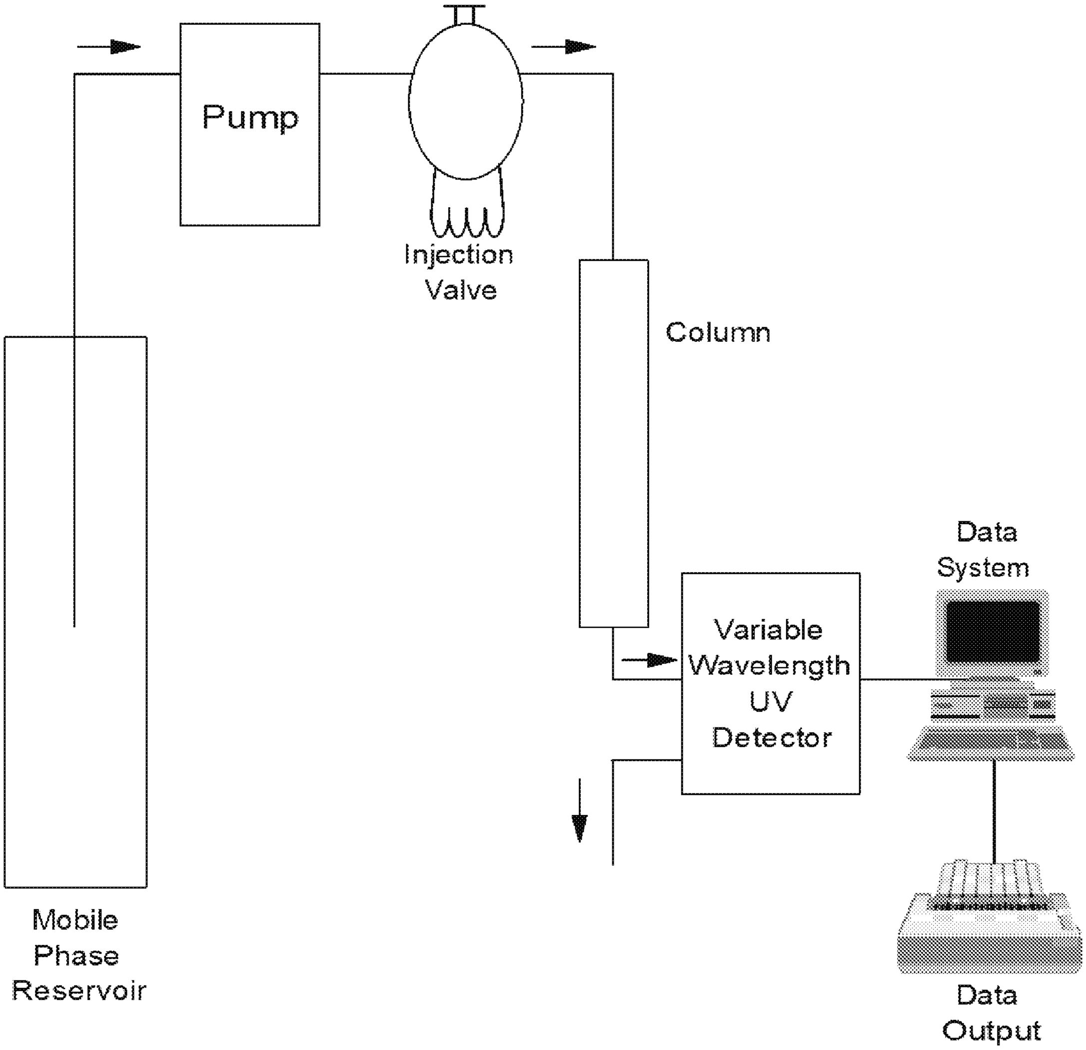

Figure 10-4. HPLC System

10.5 Metals Using an Inductively Coupled Argon Plasma Mass Spectrometry Analytical System

Upon receipt from the field, the samples are checked against the COC forms and then logged into the LIMS system. Each sample component is examined to determine if damage occurred during travel. Color, appearance, and other sample particulars are noted. Sample preparation and analysis procedures are based on EPA Compendium Methods IO-3.1[22] and IO-3.5[6], respectively for the Determination of Metals in Ambient Particulate Matter using ICP-MS techniques. A complete description of the preparation and analytical procedures are presented in the SOPs for quartz and glass fiber (8x10") filter prep (ERG-MOR-084) and for Teflon 47mm filter prep (ERG-MOR-085) and analysis (ERG_MOR-095) in Appendix C. These procedures were approved as NAAQS Federal Equivalency Methods (FEM) for the analysis of Lead for Total Suspended Particulate (TSP) on quartz and glass fiber filters (EQL-0512-201[7]) and for PM_{10} on Teflon filters (EQL-0512-202[8]). Analysis hold time for metals filters is 180 days.

The ICP-MS consists of an inductively coupled plasma source, ion optics, a quadrupole MS, a recirculator and an autosampler. The MS will be mass calibrated and resolution checked. Resolution at low mass is indicated by magnesium isotopes 7Li, 24, 25, and 26Mg, 59Co, 115In, 206, 207, and 208Pb and U238. Instrument stability must be demonstrated by running a tuning (daily performance check) solution [1 micrograms per liter (µg/L) of barium, bismuth, cerium, cobalt, indium, lead, lithium and uranium, and 15 µg/L of magnesium] 10 times with a resulting Relative Standard Deviation (RSD) of absolute signals for all analytes less than 2 or 5 percent, depending on element and instrument acquisition mode. Sample and waste disposal procedures are outlined in ERG-MOR- 033, the *SOP for Hazardous Waste*.

10.6 Hexavalent Chromium Analytical System

Hexavalent chromium filter samples are stored in the freezer after they are received from the field prior to analysis. Internal studies have shown that the hexavalent chromium does not degrade for up to 21 days if the samples are stored in the freezer before extraction. Upon receipt from the field, the samples are checked against the COC forms and then logged into LIMS. Due to oxidation/reduction and conversion between the trivalent and hexavalent chromium, the extraction is performed immediately prior to analysis. Therefore, it is important that the IC be equilibrated, calibrated and ready for analysis before filters are extracted. Sample preparation is performed by removing the filter from the filter holder and placing it into a 14 mL polystyrene tube. The filters are extracted in 10 mL of a 20 millimolar (mM) sodium bicarbonate solution. The tubes are shaken for 45 minutes using a wrist action shaker before a 2.5 mL aliquot is removed for analysis on the IC. All analysis is completed within 24 hours of the filter extraction.

The analytical separation for the hexavalent chromium is performed using a Dionex-600 IC or Dionex ICS-5000 with a Dionex LC 20 Chromatography Enclosure with a post-column reagent delivery device and an advanced gradient pump configured with an IonPac AS7 analytical column and an IonPac NG1 guard column. Both of ERG's ICs use the Dionex Chromeleon® data system. For the Dionex-600 IC, samples are injected using a Dionex AS40 autosampler. The samples analyzed with the Dionex ICS-5000 are injected using an AS-DV autosampler. A mobile phase is used to perform the analytical separation at a flow rate of 1.0 mL/min, and a post-column reagent flow rate of 0.3 mL/min. The multiwavelength UV detector is set at 530 nm. The samples are prepped and analyzed following ASTM D7614-12[9] method and the *SOP for the Preparation and Analysis of Ambient Air for Hexavalent Chromium by Ion Chromatography* (ERG-MOR-063) that is presented in Appendix C. Sample and waste disposal procedures are outlined in ERG-MOR- 033, the *SOP for Hazardous Waste*.

SECTION 11
QUALITY CONTROL REQUIREMENTS

This section describes the quality control requirements for each of the major program components (NMOC, SNMOC, VOC, Carbonyls, PAMS, HAPs – SVOC, Metals and Hexavalent Chromium). As there is not a current need for some of the HAPS (SVOC analysis following TO-13A(10)/SW 846 Method 8270E(11), PCB/Pesticides(13), inorganic acids(14), etc.), this information is not provided. As soon as these analyses are requested by EPA or States, however, the QAPP will be modified and a new set of MDLs will be completed and presented to EPA. The 2018 MDLs are presented in this section.

11.1 Sample Canister Integrity Studies

Before any SNMOC or VOC samples are collected for a program, all stainless-steel sample canisters are checked for leaks. The canisters are evacuated to less than 25" Hg. The canister vacuum, measured on a Heise gauge, and the barometric pressure is recorded. After 7 days, the canister vacuum and barometric pressure is remeasured. The canisters are considered leak-free if there is less than 1" Hg difference in vacuum (adjusted for differences in the barometric pressure). The canisters are then cleaned using the procedure described in Section 10. For the canister to be used without further cleanup, an analysis must show that it meets the quality objective for cleanliness.

11.2 Standard Traceability

The standards used for all analytes are vendor-supplied National Institute of Standards and Technology (NIST) standards or vendor-supplied referenced to a NIST standard. All analytical methods are also certified by comparison to a second source NIST-traceable standard. The ERG-MOR-022 *SOP for the Preparation of Standards in the ERG Laboratory*, provides direction for preparing standards from solid or liquid chemicals. The SOP used to prepare canister standards is *SOP for Standard Preparation Using Dynamic Flow Dilution System*, ERG-MOR-061 (Appendix C).

11.3 Accuracy and Acceptance

As ambient air measurements encompass a range of compounds and elements whose individual concentrations are unknown, defining absolute accuracy is not possible. Instead, accuracy is determined by comparing the analysis of duplicate samples and of standards of known concentration. The criteria for the analysis of duplicate (or collocated) samples and their replicate analyses are found in Section 4. Accuracy of analysis is based on the accuracy of the calibration, including the accuracy of the calibration standards. Each instrument calibration is discussed by method in Section 13 of this QAPP. Accuracy is monitored throughout the program using QC samples. Required QC samples and their criteria and corrective actions are discussed by the methods listed below.

11.3.1 SNMOC Analysis

Prior to sample analysis for SNMOC, a continuing calibration verification (CCV) standard of hydrocarbons, prepared using either a NIST-traceable Linde or Air Environmental high pressure gas, is analyzed daily to ensure the validity of the current Response Factors (RF). This standard will have an approximate concentration range from 5 ppbC to 400 ppbC. The concentrations are compared to the calculated theoretical concentrations of the CCV. The standard analysis is considered acceptable if the percent recovery is 70-130 percent for 10 selected compounds.

If the CCV does not meet the percent recovery criterion, a second CCV is analyzed. If the second CCV meets the criterion, the analytical system is considered in control. If the second CCV does not meet acceptance criteria, a leak test and system maintenance are performed. Following these maintenance procedures, a third CCV analysis can be performed. If the criterion is met by the third analysis, the analytical system is considered in control. If maintenance causes a change in system response, a new calibration curve is required.

A system blank of cleaned, humidified N_2 is analyzed after the CCV and before the sample analysis. The system is considered in control if the total NMOC concentration for the system blank is less than or equal to 20 ppbC.

CCV requirements are presented in Table 11-1. If both the hydrocarbon and TO-15[(4)] parameters are requested from same sample, the instrument must conform to the standard QC procedures listed in both Tables 11-1 and 11-2 (for VOC QC requirements).

11.3.2 VOC Analysis

The tune of the GC/MS is verified using a 4-Bromofluorobenzene (BFB) instrument performance check sample daily. The acceptance criteria for the BFB are presented in Table 11-3. The internal standards for this method are hexane-d_{14}, 1,4-difluorobenzene, and chlorobenzene-d_5. The internal standard responses must be evaluated to ensure instrument stability throughout the day.

Before sample analyses, a standard prepared at approximately 2.5 ppbV from a NIST-traceable Linde or Air Environmental gas cylinder is used for a CCV. The resulting response factor for each compound is compared to the average calibration curve response factors generated from the GC/MS using the Agilent ChemStation® Software. Correspondence within an absolute value of less than or equal to 30 percent difference is considered acceptable for the quantitated compounds. If the first CCV does not meet this criterion, a second CCV will be analyzed. If the second CCV is acceptable, sample analysis can continue. If the second CCV does not meet acceptance criteria, then a leak check and system maintenance are performed. If the system maintenance is completed and a third CCV analysis meets the criterion, then analysis may continue. If the maintenance causes a change in the system response, a new calibration curve must be analyzed before sample analyses can begin.

Table 11-1
Summary of SNMOC Quality Control Procedures

QC Check	Frequency	Acceptance Criteria	Corrective Action
Multiple point calibration (5 points minimum); propane, hexane, benzene, octane, and decane bracketing the expected sample concentration. Laboratory Control Standard (LCS) (or Initial Calibration Verification (ICV))	Quarterly	Average Response Factor (RF) curve fit with RF RSD within ±20% ICV Recovery for selected hydrocarbons 70-130%	1) Repeat individual sample analysis 2) Prepare new calibration standards and repeat
Continuing calibration verification (CCV) using Certified Standard	Daily, prior to sample analysis	Recovery for 10 selected hydrocarbons spanning the carbon range 70-130 %	1) Repeat analysis 2) Reprepare and reanalyze 3) Repeat calibration curve
Method Blank Analysis	Daily, following calibration check	≤ 20 ppbC total	1) Repeat analysis 2) Check system for leaks 3) Reanalyze blank
Canister cleaning certification	One canister analyzed on the Air Toxics system per batch of 12	≤ 20 ppbC total	Reclean canisters and reanalyze

Table 11-2
Summary of Air Toxics Canister VOC Quality Control Procedures

QC Check	Frequency	Acceptance Criteria	Corrective Action
BFB Instrument Tune Performance Check	Daily[b], prior to sample analysis	Evaluation criteria presented in Section 16.1.1 of the SOP and Table 11-3 of this QAPP.	1) Retune 2) Clean ion source and/or quadrupole
Initial calibration (ICAL) consisting of at least 5 points bracketing the expected sample concentration.	Following any major change, repair, or maintenance or if daily QC is not acceptable. Recalibration not to exceed three months.	1) % RSD of Response Factors ≤ 30% RSD (with two exceptions of up to ± 40% for non-Tier I compounds only) 2) Internal Standard (IS) response ±40% of mean curve IS response 3) Relative Retention Times (RRTs) for target peaks ±0.06 units from mean RRT 4) IS RTs within 20 seconds of mean 5) Each calibration standard concentration must be within ±30% of nominal (for Tier I compounds)	1) Repeat individual sample analysis 2) Repeat linearity check 3) Prepare new calibration standards and repeat analysis
LCS ({ICV} Second source calibration verification check)	Following the calibration curve	The response factor ≤ 30% Deviation from calibration curve average response factor	1) Repeat calibration check 2) Repeat calibration curve
Continuing Calibration Verification (CCV) of approximately mid-point of the calibration curve[a] using a Certified Standard	Before sample analysis on the days of sample analysis [b]	The response factor ≤ 30% Deviation from the calibration curve average RRF (Relative Response Factor)	1) Repeat calibration check 2) Repeat calibration curve

[a] The same QA criteria are needed for SNMOC and PAMS analysis.
[b] Every 24 hours frequency.

Table 11-2
Summary of Air Toxics Canister VOC Quality Control Procedures (Continued)

QC Check	Frequency	Acceptance Criteria	Corrective Action
Method Blank Analysis (Zero Air or N_2 Sample Check)	Daily[b], following BFB and calibration check; prior to sample analysis	1) <3x MDL or 0.2 ppbV, whichever is lower 2) IS area response ± 40% and IS RT ± 0.33 min. of most recent ICAL	1) Repeat analysis with new blank canister 2) Check system for leaks, contamination 3) Reanalyze blank
Duplicate and Replicate Analysis	All duplicate and collocate field samples	<25% RPD for compounds greater than 5 x MDL	1) Repeat sample analysis 2) Flag data in LIMS; Flag in AQS as permitted
Canister Cleaning Certification	One canister analyzed on the Air Toxics system per batch of 12	<3x MDL or 0.2 ppbV, whichever is lower	Reclean canisters and reanalyze
Preconcentrator Leak Check	Each standard and sample canister connected to the preconcentrator/ autosampler	≤ 0.2 psi change/minute	1) Retighten and reperform leak check 2) Provide maintenance 2) Re-perform leak check test

[a] The same QA criteria are needed for SNMOC and PAMS analysis.
[b] Every 24 hours frequency.

Table 11-2
Summary of Air Toxics Canister VOC Quality Control Procedures (Continued)

QC Check	Frequency	Acceptance Criteria	Corrective Action
Sampler Certification - Standard Challenge with a reference can and a Zero Check with a reference can	Annual	Challenge: Within 15% of the concentration in the reference canister. Zero: up to 0.2 ppbV or 3x MDL (whichever is lower) higher than the reference can	1) Repeat certification of samplers, a requirement for Tier I compounds 2) Notify Program Manager (flagging non-Tier I compound data for sampler may be an option)
Sampling Period	All samples	24 hours ± 1 hours	1) Notify Program Manager 2) Flag samples 22-23 hours and 25-26 hours in AQS with a "Y" flag 3) Invalidate and re-sample for > 24±2 hours
Retention Time (RT)	All qualitatively identified compounds	RT within ± 0.06 RRT units of most recent initial calibration average RT	Repeat analysis
Samples – Internal Standards	All samples	IS area response within ± 40% and IS RT within ± 0.33 min. of most recent calibration average IS response	Repeat analysis

[a] The same QA criteria are needed for SNMOC and PAMS analysis.
[b] Every 24 hours frequency.

Table 11-3. BFB Key Ion Abundance Criteria

Target Mass	Rel. To Mass	Lower Limit %	Upper Limit %
50	95	8	40
75	95	30	66
95	95	100	100
96	95	5	9
173	174	0	2
174*	95	50	120
175	174	4	9
176	174	93	101
177	176	5	9

* alternate base peak

After acceptable analysis of the daily standard has been demonstrated, a system blank consisting of clean, humidified air or N_2 is analyzed. A concentration per compound of < 3x MDL or 0.2 ppbV, whichever is lower (as outlined in Table 11-2) indicates that the system is in control. If a concentration greater than the acceptance criterion is detected, a second system blank is analyzed. If the second system blank fails, system maintenance is performed. Another system blank can be analyzed and if it is in control, ambient air samples are analyzed. All other QC procedure acceptance criteria and corrective actions are presented in Table 11-2.

11.3.3 Carbonyl Compounds Analysis

Daily CCVs prepared from NIST traceable stocks are performed to ensure that the analytical procedures are in control. CCVs are performed every 12 hours or less when samples are analyzed. Compound responses in the CCVs must have a percent recovery between 85-115 percent. Compound retention time drifts are also measured from this analysis and tracked to ensure that the HPLC instruments are operating within acceptable parameters.

If one of these CCV does not meet the criterion, analysis of a second injection of the CCV is performed. If the second CCV does not pass or if more than one CCV does not meet the criterion, a new standard is prepared and analyzed. If it fails a third time, a new calibration curve (at least 5 concentration levels) is analyzed. All samples analyzed with the unacceptable CCV will be reanalyzed.

Crotonaldehyde tautomerizes into two chromatographically separate peaks after it is spiked onto the DNPH cartridge. The best analytical recovery for crotonaldehyde is determined when both the peaks are integrated together for all samples and QC.

Acetaldehyde elutes with its stereoisomer. The best analytical recovery for acetaldehyde is determined when both peaks are integrated together for all samples and QC.

Acetonitrile system blanks (or solvent blanks) bracket each sequence, with one at the beginning of the sequence and one at the end. The system is considered in control if target compound concentrations are less than the current laboratory MDLs. Quality procedures determined for the carbonyl analysis ensure that ambient air samples are collected in the prescribed manner and that compound quantitative analyses are performed with known bias and precision. The quality procedures for carbonyl analysis are presented in Table 11-4.

11.3.4 PAH Analysis

Every 12 hours, the mass spectrometer used for PAH analysis must have an acceptable Decafluorotriphenylphosphine (DFTPP) instrument performance tune check meeting the criteria listed in Table 11-5 when 1 µL or less of the GC/MS tuning standard, depending on instrument sensitivity, is injected through the GC (50 nanogram (ng) on column).

Samples should be received with filters folded and inserted into the glass thimble cartridge with the sorbent media. It will be noted on the COC and extraction bench sheet if a filter is received in a petri dish, instead of a glass thimble. Prior to sample analyses, a daily CCV must be analyzed, usually a standard prepared at approximately the midpoint of the calibration curve from NIST-traceable PAH stock solution. The resulting response factor for each

Table 11-4
Summary of Carbonyl Quality Control Procedures

Parameter	QC Check	Frequency	Acceptance Criteria	Corrective Action
HPLC Efficiency	Analyze Second Source QC (SSQC) sample	Once per 12 hours or less	1) Resolution between acetone and propionaldehyde ≥ 1.0 2) Column efficiency > 5,000 plate counts	1) Eliminate dead volume 2) Back flush 3) Replace the column repeat analysis
DNPH Peak	All samples	Every chromatogram from an extracted cartridge (field sample, method blank, lot blank, and BS/BSD)	DNPH must be ≥ 50% of the DNPH are in the laboratory QC samples	1) Sample concentration will be flagged as estimate ("E")
Sampler Certification	Zero Challenge cartridge with a reference cartridge	Annual	Each compound must be ≤ 0.2 ppbV above the reference cartridge	1) Repeat certification of samplers, a requirement for Tier I compounds 2) Notify Program Manager (flagging non-Tier I compound data for sampler may be an option)
ICAL	Run a 5-point calibration curve	At setup or when calibration check is out of acceptance criteria (at least every 6 months)	1) Correlation coefficient at least 0.999, relative error for each level against calibration curve ≤ 20% 2) The absolute value of the intercept/slope of the calibration curve must be less than the MDL for each compound	1) Check integration 2) Reanalyze 3) Reprepare standards and recalibrate
ICV	Analyze SSQC sample	After calibration in triplicate	85-115% recovery	1) Check integration 2) Recalibrate 3) Reprepare standard

Table 11-4
Summary of Carbonyl Quality Control Procedures (Continued)

Parameter	QC Check	Frequency	Acceptance Criteria	Corrective Action
Retention Time	Analyze SSQC	Once per 12 hours or less	Each target compound within ± 2.5% of the mean calibration standards RT (set in Agilent® software)	1) Check integration, 2) Check for plug in LC 3) Check column temperature in LC
CCV	Analyze SSQC sample	Once per 12 hours or less	85-115% recovery	1) Check integration 2) Reanalyze, reprepare standard, or recalibrate 3) Reanalyze samples not bracketed by acceptable standard
Solvent Blank (aka Continuing calibration blank (CCB), System Blank, or Laboratory Reagent Blank (LRB))	Analyze acetonitrile	Bracket sample batch, 1 at beginning and 1 at end of batch	Measured concentration must be < MDL for each compound	1) Locate contamination and correct 2) Flag associated data
Sampling Period	All samples	All samples	24 hours ± 1 hours	1) Notify Program Manager 2) Flag samples 22-23 hours and 25-26 hours in AQS with a "Y" flag 3) Invalidate and re-sample for > 24±2 hours

Table 11-4
Summary of Carbonyl Quality Control Procedures (Continued)

Parameter	QC Check	Frequency	Acceptance Criteria	Corrective Action
Lot Blank Check	Analyze blank for new lots received	Analyze 1.0 % of total lot or a minimum of 3 cartridges, whichever is greater	Compounds must be less than values listed: Formaldehyde <0.15 µg/cartridge (0.03 µg/mL) Acetaldehyde <0.10 µg/cartridge (0.02 µg/mL) Acetone <0.30 µg/cartridge (0.06 µg/mL) Others <0.10 µg/cartridge (0.02 µg/mL)	1) Reanalyze an additional set of cartridges from the new lot 2) Notify vendor if lot blank continues to fail and acquire new lot if possible 3) Flag data associated with bad lot
Extraction Solvent Method Blank (ESMB)	Aliquot of extraction solvent prepared with samples during extraction	First extraction per month and when acetonitrile lot changes	All target compounds must be < MDL	1) Check integration 2) Reanalyze 3) Locate and resolve contamination in extraction glassware/solvent 4) Flag batch data
Field Blank (FB) Check	Field blank samples collected in the field	Monthly (if provided by site)	Underivatized compound concentrations must be less than values listed: Formaldehyde <0.3 µg/cartridge (0.06 µg/mL) Acetaldehyde <0.4 µg/cartridge (0.08 µg/mL) Acetone <0.75 µg/cartridge (0.15 µg/mL) Others <7.0 µg/cartridge (1.4 µg/mL)	1) If FB fails, notify site coordinator, schedule another FB. Additional FBs are collected until the problem is corrected and data are acceptable 2) Flag samples since the last acceptable FB

Table 11-4
Summary of Carbonyl Quality Control Procedures (Continued)

Parameter	QC Check	Frequency	Acceptance Criteria	Corrective Action
Duplicate or Collocate Samples	Analysis of duplicate and collocated samples	As collected (10% of sampling schedule)	≤ 20% RPD for concentrations ≥ 0.5 µg/cartridge	1) Check integration 2) Check instrument function 3) Reanalyze duplicate samples 4) Flag data in LIMS (and AQS as permitted)
Replicate Analyses	Replicate injections	One per batch. Performed on every duplicate and collocate sample or if none available, on a field sample	≤ 10% RPD for concentrations ≥ 0.5 µg/cartridge	1) Check integration 2) Check instrument function 3) Reanalyze sample
MB (BLK)	Analyze MB	One per batch of 20 samples	Underivatized compound concentrations must be less than values listed: Formaldehyde <0.15 µg/cartridge (0.03 µg/mL) Acetaldehyde <0.10 µg/cartridge (0.02 µg/mL) Acetone <0.30 µg/cartridge (0.06 µg/mL) Others <0.10 µg/cartridge (0.02 µg/mL)	1) Reanalyze MB 2) Check extraction procedures 3) Flag batch data

Table 11-4
Summary of Carbonyl Quality Control Procedures (Continued)

Parameter	QC Check	Frequency	Acceptance Criteria	Corrective Action
Blank Spike/Blank Spike Duplicate, (BS/BSD or LCS/LCSD)	Analyze BS/BSD (or LCS/LCSD)	One BS/BSD (LCS/LCSD) per batch of 20 samples	80-120% recovery for Formaldehyde and Acetaldehyde and 70-130% for all other compounds. BSD (LCSD) precision ≤20% RPD of BS (LCS)	1) Reanalyze BS/BSD (LCS/LCSD) 2) Check calibration 3) Check extraction procedures

Note: Crotonaldehyde tautomerizes into two chromatographically separate peaks after it is spiked onto the DNPH cartridge. The best analytical recovery is determined when both peaks are integrated together for all samples and QC. Acetaldehyde elutes with its stereoisomer. The best analytical recovery for Acetaldehyde is determined when both peaks are integrated together for all samples and QC. Breakthrough cartridges are not submitted or analyzed as specified by Compendium Method TO-11A.

compound will be compared to the average calibration curve response factors. Correspondence within an absolute value of less than or equal to 30 percent difference is considered acceptable. If the first CCV does not meet this criterion, a second CCV will be analyzed. If the second CCV is acceptable, sample analysis can continue. If the second CCV does not meet acceptance criteria, then a leak check and system maintenance are performed. If the system maintenance is completed and a third CCV analysis meets the criterion, then analysis may continue. If the maintenance causes a change in the system response, a new calibration curve must be analyzed before sample analyses can begin.

EPA Compendium Method TO-13A[10] employs and spikes two different types of surrogates. The Field Surrogates, fluoranthene-d_{10} and benzo(a)pyrene-d_{12}, are spiked onto the PUF media prior to shipment to the field; acceptable recoveries for these field surrogates are in the range of 60 to 120 percent. The laboratory surrogates, fluorene-d_{10} and pyrene-d_{10}, are spiked into the PUF immediately before extraction; acceptable recoveries for these laboratory surrogates are 60 to 120 percent.

Table 11-5. DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	10 to 80% of base peak
68	< 2% of mass 69
69	Present
70	< 2% of mass 69
127	10 to 80% of base peak
197	< 2% of mass 198
198	Base peak (100% relative abundance) or >50% of mass 442
199	5 to 9% of mass 198
275	10 to 60% of base peak
365	> 1.0% of mass 198
441	Present but < 24% of mass 442
442	Base peak, or >50% of mass 198
443	15 to 24% of mass 442

Note: All ion abundances must be normalized to the nominal base peak, 198 or 442. This criterion is based on the tune criteria for Method 8270D.

Internal standard responses and retention times must also be evaluated for stability. The SIM procedures of EPA Compendium Method TO-13A[(10)] preclude the use of guidelines for qualitative analysis of mass spectra, since complete mass spectra are not acquired when SIM procedures are used. Quantitative analysis for each compound is performed relative to the assigned internal standard. The following internal standard assignments are suggested for PAH analysis are presented in Table 11-6. All method criteria and MQOs for ERG's PAH analysis are listed in Table 11-7.

Table 11-6. Internal Standards and Associated PAHs

Internal Standard	Associated Compound
Naphthalene-d_8	Naphthalene
Acenaphthelene-d_{10}	Acenaphthylene Acenaphthene Fluorene 9-Fluorenone Pyrene Retene Fluoranthene
Phenanthrene-d_{10}	Phenanthrene Anthracene
Chrysene-d_{12}	Cyclopenta(c,d)pyrene Benz(a)anthracene Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(e)pyrene Benzo(a)pyrene Chrysene
Perylene-d_{12}	Perylene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene Coronene

Table 11-7
Summary of Quality Control Procedures for Analysis of SVOC Samples for PAHs

Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
DFTPP instrument tune check	Daily prior to calibration check and sample analysis; every 12 hours if instrument is operated 24 hours/day	Evaluation criteria presented in Section 11, Table 11-5	1) Re-analyze 2) Prepare new tune check standard; analyze 3) Re-tune instrument; reanalyze 4) Clean ion source; re-tune instrument; reanalyze
Solvent Blank (SB)	Prior to ICAL	All target compounds < MDL	1) Reanalyze 2) Perform maintenance on GC; reanalyze
Five-point (minimum) calibration (ICAL)	Following any major change, repair, or maintenance if daily quality control check is not acceptable. Minimum frequency every six weeks	≤ 30% RSD of the RRFs for each compound; Avg Relative Response Factor (RRF) above or equal to minimum RRF limit for each pollutant; ≤ 30% the nominal concentration required for Tier I compounds RRTs within ± 0.06 RRT units of mean RRT of calibration IS RT within ± 20.0 sec of mean RT of calibration	1) Repeat individual calibration standard analyses 2) Check integrations and calculations 3) Prepare new calibration standards and repeat analysis 4) Perform maintenance on GC, especially leak check and repeat analysis 5) Clean ion source and repeat analysis

Table 11-7
Summary of Quality Control Procedures for Analysis of SVOC Samples for PAHs (Continued)

Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Retention Time (RT)	All qualitatively identified compounds and internal standard	RRT set in software to be no larger than + 0.25 minutes	Repeat analysis
Secondary Source Calibration Verification (SCV)	Immediately after each ICAL	≤ 30% Difference for each compound RRF compared to the mean RRF of the calibration curve.	1) Repeat SCV analysis 2) Check calculations 3) Prepare a new SCV standard and repeat analysis 4) Perform maintenance on GC, especially leak check; reanalyze 5) Clean ion source; reanalyze
Continuting Calibration Verification (CCV) Standard	Daily (or every 12 hours)	Above or equal to RRF minimum and ≤ 30% Difference for each compound RRF compared to the mean RRF of the calibration curve.	1) Repeat individual sample analyses 2) Check calculations 3) Prepare a new CCV standard and repeat analysis 4) Perform maintenance on GC, especially leak check; reanalyze 5) Clean ion source; reanalyze
Solvent Method Blank (SMB)	One with every extraction batch of 20 or fewer field-collected samples.	All target compounds < MDL	1) Check integration 2) Reanalyze 3) Flag samples 4) Remove solvent lot from use
Method Blank (MB)	With every extraction batch ≤ 20 samples	All analytes < 2x MDL	1) Repeat analysis 2) Flag data
Blank Spike (BS) or (LCS) BSD (or LCSD)	One BS (or LCS) with every extraction batch ≤ 20 samples. BSD (or LCSD) once per quarter.	60-120% recovery of nominal for all compounds ≤ 20% RPD compared to BS (or LCS)	1) Repeat analysis 2) Flag data

Table 11-7
Summary of Quality Control Procedures for Analysis of SVOC Samples for PAHs (Continued)

Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Surrogate compound recoveries: Laboratory surrogates fluorene-d_{10} pyrene-d_{10} Field Surrogates fluoranthene-d_{10} benzo(a)pyrene-d_{12}	Every sample/blank/BS	60-120% Recovery	1) Repeat analysis 2) Check calculation 3) Flag surrogate data 4) Flag sample data if both field or both lab surrogates fail
Internal Standard Response: naphthalene-d_8 acenaphthylene-d_{10} chrysene-d_{12} perylene-d_{12}	Every sample/blank/BS	Within 50% to 200% of the ISs in the most recent initial calibration CAL4	1) Repeat analysis 2) Invalidate or flag data if unable to reanalyze
Cartridge Lot Blank	One cartridge (and filter) for each batch of prepared cartridges for a particular sample date.	All target compounds ≤ 2 times the MDL	1) Repeat analysis 2) Invalidate or flag data if unable to reanalyze prior to cartridge shipment
Field Blank	Monthly (or as provided by site)	Target compounds ≤ 5 times the MDL	1) If FB fails, notify site coordinator, schedule another FB. Additional FBs are collected until the problem is corrected and data are acceptable 2) Flag samples since the last acceptable FB when input in AQS
Replicate Analysis	Replicate sample, on each collocate or at a minimum one per sequence	≤ 10% RPD for concentration ≥ 0.5 ng/µL or lowest cal point, whichever is less.	1) Check integration 2) Check instrument function 3) Reanalyze 4) Flag replicate samples

Table 11-7
Summary of Quality Control Procedures for Analysis of SVOC Samples for PAHs (Continued)

Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Collocate Samples	Collocated samples, 10% of field samples, or as collected	≤ 20% RPD for concentration ≥ 0.5 ng/µL or lowest ICAL level, whichever is less	1) Check integration 2) Check instrument function 3) Reanalyze 4) Flag collocated samples
Sampling Period	All samples	24 hours ± 1 hours	1) Notify Program Manager 2) Flag samples 22-23 hours and 25-26 hours in AQS with a "Y" flag 3) Invalidate and re-sample for > 24±2 hours

NOTE: Matrix Spikes are not performed as required by Compendium Method TO-13A. Matrix spikes are not required by ASTM D2609.

11.3.5 Metals Analysis

The mass spectrometer used for metals analysis must have an acceptable daily performance check using the tuning solution before each analysis. Daily performance checks are done in both standard and kinetic energy discrimination (KED) mode to verify instrument performance in both modes. Performance specifications are presented in Table 11-8. Analysis of the metals will be performed by ICP-MS for antimony, arsenic, beryllium, cadmium, total chromium, cobalt, lead, manganese, mercury, nickel, and selenium. The internal standards for this method are lithium, scandium, germanium, yttrium, indium, terbium, holmium, and bismuth. Internal standard responses must be evaluated for stability. Gold is added to each of the standards before analysis to prevent the loss of mercury on labware or instrument tubing in the ICP-MS.

Daily calibration, using a calibration blank and at least 5 non-zero standards prepared from NIST-traceable stock solutions, is performed to ensure that the analytical procedures are in control. To be considered acceptable, the calibration curve must have a correlation coefficient ≥ 0.998. Replicate analysis of the calibration standards must have an RSD ≤ 10 percent, except for the second calibration standard (CAL2). This standard uses the same concentrations as the Limit of Quantitation (LOQ) standard, which are near or less than that of the MDL, therefore an RSD > 10 percent is acceptable. After calibration, an Initial Calibration Verification (ICV), Initial Calibration Blank (ICB), High Standard Verification (HSV), Interference Check Standard A (ICSA), and Interference Check Standard B (ICSAB) are analyzed to ensure quality before the analysis of the samples.

If the initial calibration check does not meet criteria, a second calibration check analysis is performed. If the second set does not pass, or if one or more of the daily QC checks do not meet criteria, a new calibration curve is prepared and analyzed. All samples analyzed with the unacceptable QC check will be reanalyzed or flagged appropriately when necessary. During the analysis of the samples, the Continuing Calibration Verification (CCV) and Continuing Calibration Blank (CCB) are analyzed immediately before the analysis of samples, every 10 samples, and at the end of every analysis batch. he ICSA and ICSAB are analyzed once per analysis day. Quality procedures for metals analysis are shown in Table 11-9.

Table 11-8 Instrument Mass Calibration & Performance Specifications

Parameter	Peak Width	Sensitivity/Criteria*	RSD
iCAP-Q Criteria			
Standard Mode			
Bkg4.5	NA	< 1.0 cps	N/A
7Li	0.65–0.85	> 50,000 cps	< 2% RSD
24Mg	0.65–0.85	> 500,000 cps	< 2% RSD
25Mg	0.65–0.85	> 70,000 cps	< 2% RSD
26Mg	0.65–0.85	> 80,000 cps	< 2% RSD
59Co	0.65–0.85	> 100,000 cps	< 2% RSD
115In	0.65–0.85	> 220,000 cps	< 2% RSD
206Pb	0.65–0.85	> 70,000 cps	< 2% RSD
207Pb	0.65–0.85	> 60,000 cps	< 2% RSD
208Pb	0.65–0.85	> 100,000 cps	< 2% RSD
238U	0.65–0.85	> 300,000 cps	< 2% RSD
140Ce16O/140Ce	NA	< 0.02	N/A
137Ba++/137Ba+	NA	< 0.03	N/A
Bkg220.7	NA	< 2.0 cps	N/A
Analyzer Pressure	NA	< 10^{-6} mbar	NA
KED Mode†			
Bkg4.5	NA	< 0.5 cps	N/A
24Mg	0.65–0.85	> 3,000 cps	< 5% RSD
25Mg	0.65–0.85	> 500 cps	< 5% RSD
26Mg	0.65–0.85	> 600 cps	< 5% RSD
59Co	0.65–0.85	> 30,000 cps	< 2% RSD
115In	0.65–0.85	> 30,000 cps	< 2% RSD
206Pb	0.65–0.85	> 60,000 cps	< 2% RSD
207Pb	0.65–0.85	> 50,000 cps	< 2% RSD
208Pb	0.65–0.85	> 80,000 cps	< 2% RSD
238U	0.65–0.85	> 80,000 cps	< 2% RSD
140Ce16O/140Ce	NA	< 0.01	N/A
59Co/35Cl16O	NA	> 18.0	N/A
Bkg220.7	NA	< 2.0 cps	N/A

*cps – Counts per second
† – There are no vacuum requirements for KED mode

Table 11-8 Instrument Mass Calibration & Performance Specifications (Continued)

Parameter	Peak Width	Sensitivity/Criteria*	RSD
iCAP-RQ Criteria			
Standard Mode			
Bkg4.5	NA	< 1.0 cps	N/A
7Li	0.65–0.85	> 55,000 cps	< 2% RSD
24Mg	0.65–0.85	> 500,000 cps	< 2% RSD
25Mg	0.65–0.85	> 80,000 cps	< 2% RSD
26Mg	0.65–0.85	> 100,000 cps	< 2% RSD
59Co	0.65–0.85	> 100,000 cps	< 2% RSD
115In	0.65–0.85	> 240,000 cps	< 2% RSD
206Pb	0.65–0.85	> 80,000 cps	< 2% RSD
207Pb	0.65–0.85	> 70,000 cps	< 2% RSD
208Pb	0.65–0.85	> 160,000 cps	< 2% RSD
238U	0.65–0.85	> 330,000 cps	< 2% RSD
140Ce16O/140Ce	NA	< 0.02	N/A
137Ba++/137Ba+	NA	< 0.03	N/A
Bkg220.7	NA	< 2.0 cps	N/A
Analyzer Pressure	NA	< 10^{-6} mbar	NA
KED Mode†			
Bkg4.5	NA	< 0.5 cps	N/A
24Mg	0.65–0.85	> 10,000 cps	< 5% RSD
25Mg	0.65–0.85	> 2,000 cps	< 5% RSD
26Mg	0.65–0.85	> 3,000 cps	< 5% RSD
59Co	0.65–0.85	> 30,000 cps	< 2% RSD
115In	0.65–0.85	> 35,000 cps	< 2% RSD
206Pb	0.65–0.85	> 100,000 cps	< 2% RSD
207Pb	0.65–0.85	> 90,000 cps	< 2% RSD
208Pb	0.65–0.85	> 200,000 cps	< 2% RSD
238U	0.65–0.85	> 85,000 cps	< 2% RSD
140Ce16O/140Ce	NA	< 0.01	N/A
59Co/35Cl16O	NA	> 18.0	N/A
Bkg220.7	NA	< 2.0 cps	N/A

*cps – Counts per second
† – There are no vacuum requirements for KED mode

Table 11-9
Summary of Quality Control Procedures for Metals Analysis

Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Daily Performance Check (DPR) STD Mode	Before each analysis	See Table 11-8	1) Repeat analysis of DPR 2) Re-optimize instrument tuning parameters 3) Reprepare DPR standard 4) Perform instrument maintenance
Daily Performance Check (DPR) KED Mode	Before each analysis	See Table 11-8	1) Repeat analysis of DPR 2) Re-optimize instrument tuning parameters 3) Reprepare DPR standard 4) Perform instrument maintenance
Initial Calibration Standards (IC)	Daily before each analysis, at least 5 non-zero calibration points and a blank	Correlation coefficient (R) $\geq$ 0.998 & replicate %RSD $\leq$ 10. RSDs > 10% are acceptable for the target elements in the CAL2 standard (at LOQ concentration).	1) Repeat analysis of calibration standards 2) Reprepare calibration standards and reanalyze
ICV	Immediately after calibration	Recovery 90-110%	1) Repeat analysis of ICV 2) Reprepare ICV standard 3) Recalibrate and reanalyze
ICB	Immediately after ICV	Absolute value must be < MDL	1) Locate and resolve contamination problems before continuing 2) Reanalyze or recalibrate failing elements for the entire analysis when appropriate
HSV	After ICB and before ICS	Recovery from 95-105%	1) Repeat analysis of HSV 2) Reprepare HSV
ICSA/IFA	Following the HSV	Within ±3 times LOQ from zero or from the stock standard background contamination when present	1) Repeat analysis of ICSA 2) Reprepare ICSA and analyze 3) Recalibrate or flag failing elements as necessary

Table 11-9
Summary of Quality Control Procedures for Metals Analysis (Continued)

Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
ICSAB/IFB	Following each ICSA	Recovery 80-120% of true value plus standard background contamination when present	1) Repeat analysis of ICSAB 2) Reprepare ICSAB and analyze 3) Recalibrate or flag failing elements as necessary
CCV	Analyze before samples, after every 10 samples, and at the end of each run	Recovery 90-110%	1) Reanalyze CCV 2) Reprepare CCV 3) Recalibrate and reanalyze samples since last acceptable CCV
Low Calibration Verification (LCV)	After the first and last CCV	Recovery 70-130% for Pb only	1) Reanalyze LCV 2) Reprepare LCV 3) Recalibrate and reanalyze samples since last acceptable LCV
CCB	Analyzed after each CCV	Absolute value must be < MDL	1) Reanalyze CCB 2) Reanalyze samples since last acceptable CCB
Laboratory Reagent Blank (LRB)/Blank (BLK1)	1 per batch of ≤ 20 samples	Absolute value must be < MDL	1) Reanalyze for verification 2) If > 5x MDL, failing elements for all batch QC and samples must be flagged 3) When enough sample filter remains (for quartz and glass fiber filters), a reextraction and analysis of the batch should be considered
MB/BLK2	1 per batch of ≤ 20 samples	Absolute value must be < MDL.	Flag the failing elements in the MB. Note: This QC sample is not required by the IO-3.5 method and there is no further corrective action
Standard Reference Material (SRM)	1 per batch of ≤ 20 samples	Recovery 80-120% for Pb only	1) Reanalyze 2) Flag sample data 3) Re-extract batch if possible

Table 11-9
Summary of Quality Control Procedures for Metals Analysis (Continued)

Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
LCS/BS (and LCSD/BSD for 47mm Teflon filters only)	1 per batch of ≤ 20 samples	Recovery 80-120%, ≤ 20% RPD for BS/BSD	1) Reanalyze 2) Flag data if recovery for only one or two elements fail criteria 3) Reprepare sample batch if recovery for most elements fail criteria, when possible
Duplicate (DUP1) (Laboratory Duplicate)	1 per batch of ≤ 20 samples, for quartz/TSP/Glass fiber filters only	≤ 20% RPD for sample and duplicate values ≥ 5x MDL	1) Check for matrix interference in the case of DUP1. 2) Repeat duplicate analysis 3) Flag data
Replicate Analysis (Analytical Duplicate)	1 per batch of ≤ 20 samples	≤ 20% RPD for sample and duplicate values ≥ 5x MDL	1) Repeat replicate analysis 2) Flag data
Collocated Samples (C1/C2)	10% of samples annually (for sites conducting collocated sampling)	≤ 20% RPD of samples and collocated values ≥ 5x MDL	1) Repeat C1 and/or C2 analyses if replicate analyses fail 2) Flag C1 and C2 data if associated replicate reanalyses verify failure
Matrix Spike (MS) and Matrix Spike Duplicate (MSD) for 8x10" Quartz/TSP/Glass fiber filters only	1 per batch of ≤ 20 samples	Recovery 80-120% when the parent sample concentration is less than 4 times the spike concentration. Not applicable to Teflon method	1) Flag data if recovery for only one or two elements fail criteria, or when a matrix interference is confirmed by Serial Dilution (SRD) and/or Post Digestion Spike (PDS) results 2) Reanalyze 3) Reprepare sample batch if recovery for most elements fail criteria or contamination is evident 4) Sb failures must be flagged on MS/MSD and all samples

Table 11-9
Summary of Quality Control Procedures for Metals Analysis (Continued)

Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
MS/MSD RPD for 8x10" Quartz/TSP/Glass filters only	1 per batch of ≤ 20 samples	RPD ≤20% Not applicable to Teflon method	1) Check for 4x spike concentration and non-homogenous matrix, flag as necessary 2) Reanalyze for verification
PDS	1 per batch of ≤ 20 samples	Recovery 75%-125%	1) Flag failed elements for parent sample and PDS 2) Reprepare PDS if preparation issue is suspected reason for failure
SRD	1 per batch of ≤ 20 samples	10% RPD of undiluted sample if the element concentration is ≥ 25x MDL	1) Reprepare dilution if preparation issue is suspected reason for failure 2) Flag failed analytes
Field Blank	All Field Blanks as received from field	<5x MDL	1) Flag failed elements in FB
Internal Standards (ISTD)	Every Calibration, QC and Field Sample	Recovery 60-125% of the measured intensity of the calibration blank	1) If drift suspected, stop analysis and determine cause, recalibrate if necessary 2) Reprepare sample 3) If recovery > 125% due to inherent ISTD, dilute sample and reanalyze
Sampling Period	All samples	24 hours ± 1 hours	1) Notify Program Manager 2) Flag samples 22-23 hours and 25-26 hours in AQS with a "Y" flag 3) Invalidate and re-sample for > 24±2 hours

11.3.6 Hexavalent Chromium Analysis

CCVs prepared from NIST-traceable stocks are performed each analysis day to ensure that the analytical procedures are in control. During the analysis of the samples, the ICV and ICB are analyzed immediately before the analysis of samples, a CCV and CCB after every ten injections, and at the end of every analysis batch. The acceptance criteria are between 90-110 percent recovery for the ICVs and CCVs and less than MDL for the ICBs and CCBs.

If these daily CCVs (and/or CCBs) do not meet the criterion, a second analysis of the same standard is performed. If the second CCV does not pass or if more than one daily CCV does not meet the criterion, a new standard is prepared and analyzed. If it fails a third time, a new calibration curve (with at least 5 concentration levels) is analyzed. All samples analyzed with the unacceptable CCV will be reanalyzed. The quality procedures for hexavalent chromium analysis are presented in Table 11-10.

Table 11-10
Summary of Quality Control Procedures for Hexavalent Chromium

QC Check	Frequency	Acceptance Criteria	Corrective Action
Initial 6-point calibration standards	Before every sequence	Correlation coefficient ≥ 0.995; Relative Error (RE) < 20%	1) Repeat analysis of calibration standards 2) Reprepare calibration standards and reanalyze
ICV	Before every sequence, following the initial calibration	Recovery 90-110%	1) Repeat analysis of initial calibration verification standard 2) Repeat analysis of calibration standards 3) Reprepare calibration standards and reanalyze
ICB	One per batch, following the ICV	Analyte must be < MDL	1) Reanalyze 2) Reprepare blank and reanalyze 3) Correct contamination and reanalyze blank 4) Flag data of all samples in the batch
CCV	Every 10 injections and at the end of the sequence	Recovery 90-110%	1) Repeat analysis of CCV 2) Reprepare CCV 3) Flag data bracketed by unacceptable CCV
Laboratory Control Sample (LCS/LCSD)	Two per sample batch of ≤ 20 samples	Recovery 90-110%	1) Reanalyze 2) Reprepare standard and reanalyze 3) Flag data of all samples since the last acceptable LCS
MB	One per batch	Analyte must be ≤ MDL	1) Reanalyze 2) Flag data for all samples in the batch
Replicate Analysis	Duplicate, Collocate, BS/BSD and/or replicate samples only	RPD ≤ 20% for concentrations greater than 5 x the MDL	1) Check integration 2) Check instrument function 3) Flag samples
CCB	After every CCV and at the end of the sequence	Analyte must be < MDL	1) Reanalyze 2) Reprepare blank and reanalyze 3) Correct contamination and reanalyze blank 4) Flag data of all samples in the batch

Table 11-10
Summary of Quality Control Procedures for Hexavalent Chromium (Continued)

QC Check	Frequency	Acceptance Criteria	Corrective Action
Retention Time (RT)	For identification of analyte	RT must be within 5% window of the average RT of initial calibration standards	1) Check integration/identification 2) Reanalyze
Sampling Duration	All samples	24 hours ± 1 hours	1) Notify Program Manager 2) Flag samples 22-23 hours and 25-26 hours in AQS with a "Y" flag 3) Invalidate and re-sample for > 24±2 hours

11.4 Precision

Analytical precision is estimated by repeated analysis of approximately 10 percent of the samples. The second analysis is performed in the same analytical batch as the first analysis. Duplicate and collocated samples are reanalyzed once each to determine overall precision, including sampling and analysis variability.

Precision estimates are calculated in terms of absolute percent difference. Because the true concentration of the ambient air sample is unknown, these calculations are relative to the average sample concentration.

Precision is determined as the RPD using the following calculation:

$$RPD = \frac{|X_1 - X_2|}{\overline{X}} \times 100$$

Where:

X_1 is the ambient air concentration of a given compound measured in one sample;
X_2 is the concentration of the same compound measured during duplicate/collocate/replicate analysis; and
$\overline{X}$ is the arithmetic mean of X_1 and X_2.

11.5 Completeness

Completeness, a quality measure, is calculated at the end of each year. Percent completeness is calculated as the ratio of the number of valid samples received to the number of scheduled samples (beginning with the first valid field sample received through the last field sample received). This quality measure is presented in the final report. The completeness criteria for all parameters were previously presented in Table 4-1.

Completeness is determined using the following calculation:

$$\text{Completeness} = \frac{Number\ of\ valid\ samples}{Total\ expected\ number\ of\ samples}\ x\ 100$$

11.6 Representativeness

Representativeness measures how well the reported results reflect the actual ambient air concentrations. This measure of quality can be enhanced by ensuring that a representative sampling design is employed. This design includes proper integration over the desired sampling period and following siting criteria established for each task. The experimental design for sample collection should ensure samples are collected at proper times and intervals for their designated purpose per the data quality objectives. For example, SNMOC samples are collected to gain information about PAMS volatile hydrocarbons. Therefore, collection of 3-hour samples from 6:00 a.m. to 9:00 a.m. each weekday is appropriate. Quality measures for duplicate sample collection and replicate analyses are included. ERG is not responsible for the sampling design; therefore, representativeness is beyond the scope of this QAPP. The state and local areas should designate the representativeness following EPA guidelines, however a copy of the 2018 EPA sampling schedule is presented in Appendix B.

11.7 Sensitivity (Method Detection Limits)

Based on changing EPA guidance on MDL determination procedures, the NATTS program has adopted two MDL procedures, a modified Method Update Rule (MUR) for CFR Part 136, Appendix B[19] and the Federal Advisory Committee (FAC) Single Laboratory Procedure (v2.4)[20]. In the modified MUR, the MDLs are determined using spiked sample and blank sample data, using the larger value for the new MDL. The MDLs determined from spiked samples are verified by analyzing standards at one to four times the newly determined limits. For the FAC, the historic blank sample data is used to determine the MDL and spiked samples are used if the blank data does not meet requirements. VOC, carbonyl, SVOC, metals and hexavalent chromium analyses follow one of the two methods for MDL determination.

For SNMOC and hexavalent chromium (non-NATTS program), the MDLs of the target compounds are determined by analyzing at least seven spiked samples at one concentration on the appropriate collection media (ex.- for SNMOC, 7 spiked samples in 7 individual canisters). The concentration of the spiked samples should be within five times the expected detection limit. The samples should be prepared in a minimum of three different preparation batches and analyzed over 3 non-consecutive days (minimum). This procedure follows the method listed in the 1987 CFR Part 136, Appendix B[19]. The MDLs determined from spiked samples are verified by analyzing standards at one to four times the newly determined limits.

The MDL for NMOC has not been determined in 2018. If this method is needed, a detection limit study will be performed before analysis begins. The MDLs for the SNMOC are listed in Table 11-11, for VOCs in Table 11-12, and carbonyl compounds (based on a sample volume of 1000 L) in Table 11-13. The PAH MDLs, based on a sampling volume of 300 m^3, are presented in Table 11-14.

Table 11-11. 2018 SNMOC Method Detection Limits

Target Compound	MDL (ppbC)	SQL (ppbC)	Target Compound	MDL (ppbC)	SQL (ppbC)
1,2,3-Trimethylbenzene*	0.172	0.546	Cyclopentene	0.515	1.64
1,2,4-Trimethylbenzene*	0.185	0.588	Ethane*	0.993	3.16
1,3,5-Trimethylbenzene*	0.173	0.549	Ethylbenzene*	0.096	0.305
1,3-Butadiene*	0.123	0.390	Ethylene*	2.35	7.46
1-Butene*	0.125	0.396	Isobutane*	0.051	0.161
1-Decene	0.185	0.588	Isobutene	0.131	0.417
1-Dodecene	0.611	1.943	Isopentane*	0.060	0.191
1-Heptene	0.082	0.262	Isoprene*	0.055	0.176
1-Hexene*	0.085	0.272	Isopropylbenzene*	0.089	0.284
1-Nonene	0.127	0.404	*m,p*-Xylene*	0.220	0.701
1-Octene	0.096	0.305	*m*-Diethylbenzene*	0.446	1.42
1-Pentene*	0.060	0.190	Methylcyclohexane*	0.070	0.222
1-Tridecene	0.288	0.914	Methylcyclopentane*	0.115	0.365
1-Undecene	0.390	1.24	*m*-Ethyltoluene*	0.219	0.696
2,2,3-Trimethylpentane	0.057	0.182	*n*-Butane*	0.076	0.241

* PAMS compounds
NOTE: MDL's reported are from Instrument 1. New MDLs will be reported for Instrument 4 if required.

Table 11-11. 2018 SNMOC Method Detection Limits

Target Compound	MDL (ppbC)	SQL (ppbC)	Target Compound	MDL (ppbC)	SQL (ppbC)
2,2,4-Trimethylpentane*	0.132	0.419	*n*-Decane*	0.238	0.755
2,2-Dimethylbutane*	0.084	0.267	*n*-Dodecane*	0.445	1.41
2,3,4-Trimethylpentane*	0.060	0.190	*n*-Heptane*	0.075	0.239
2,3-Dimethylbutane*	0.057	0.182	*n*-Hexane*	0.175	0.558
2,3-Dimethylpentane*	0.119	0.377	*n*-Nonane*	0.095	0.302
2,4-Dimethylpentane*	0.096	0.305	*n*-Octane*	0.062	0.197
2-Ethyl-1-butene	0.060	0.190	*n*-Pentane*	0.081	0.256
2-Methyl-1-Butene	0.089	0.283	*n*-Propylbenzene*	0.121	0.385
2-Methyl-1-Pentene	0.091	0.288	n-Tridecane	0.296	0.942
2-Methyl-2-Butene	0.287	0.912	*n*-Undecane*	0.339	1.08
2-Methylheptane*	0.199	0.631	*o*-Ethyltoluene*	0.152	0.483
2-Methylhexane*	0.136	0.431	o-Xylene*	0.131	0.417
2-Methylpentane*	0.189	0.600	*p*-Diethylbenzene*	0.191	0.609
3-Methyl-1-Butene	0.222	0.706	*p*-Ethyltoluene*	0.203	0.644
3-Methylheptane*	0.134	0.426	Propane*	0.611	1.94
3-Methylhexane*	0.262	0.833	Propylene*	0.162	0.515
3-Methylpentane*	0.075	0.239	Propyne	0.056	0.177
4-Methyl-1-Pentene	0.078	0.248	Styrene*	0.246	0.781
Acetylene*	0.044	0.139	Toluene*	0.609	1.94
Benzene*	0.080	0.255	*trans*-2-Butene*	0.036	0.114
cis-2-Butene*	0.032	0.102	*trans*-2-Hexene	0.038	0.120
cis-2-Hexene	0.063	0.200	*trans*-2-Pentene*	0.050	0.159
cis-2-Pentene*	0.055	0.175	*α*-Pinene*	0.189	0.602
Cyclohexane*	0.081	0.257	*β*-Pinene*	0.443	1.41
Cyclopentane*	0.055	0.175			

* PAMS compounds
NOTE: MDL's reported are from Instrument 1. New MDLs will be reported for Instrument 4 if required.

Table 11-12. 2018 Air Toxics Method Detection Limits

Target Compounds	MDL (µg/m³)	SQL (µg/m³)	Target Compounds	MDL (µg/m³)	SQL (µg/m³)
1,1,1-Trichloroethane	0.0750	0.238	*cis*-1,3-Dichloropropene	0.0894	0.284
1,1,2,2-Tetrachloroethane	0.144	0.457	Dibromochloromethane	0.131	0.417
1,1,2-Trichloroethane	0.104	0.330	Dichlorodifluoromethane	0.135	0.430
1,1-Dichloroethane	0.0578	0.184	Dichlorotetrafluoroethane	0.0938	0.298
1,1-Dichloroethene	0.0473	0.150	Ethyl Acrylate	0.0964	0.306
1,2,4-Trichlorobenzene	1.85	5.89	Ethyl *tert*-Butyl Ether	0.0458	0.146
1,2,4-Trimethylbenzene	0.132	0.420	Ethylbenzene	0.112	0.357
1,2-Dibromoethane	0.145	0.462	Hexachloro-1,3-Butadiene	0.293	0.931
1,2-Dichloroethane	0.0564	0.179	*m,p*-Xylene	0.157	0.498
1,2-Dichloropropane	0.0941	0.299	*m*-Dichlorobenzene	0.110	0.348
1,3,5-Trimethylbenzene	0.167	0.532	Methyl Isobutyl Ketone	0.0975	0.310
1,3-Butadiene*	0.0429	0.136	Methyl Methacrylate	0.411	1.31
Acetonitrile	0.0275	0.0873	Methyl *tert*-Butyl Ether	0.0371	0.118
Acetylene	0.0421	0.134	Methylene Chloride	0.0500	0.159
Acrolein*	0.516	1.64	*n*-Octane	0.151	0.481
Acrylonitrile	0.0232	0.0736	*o*-Dichlorobenzene	0.124	0.394
Benzene*	0.0463	0.147	*o*-Xylene	0.117	0.371
Bromochloromethane	0.0703	0.223	*p*-Dichlorobenzene	0.121	0.384
Bromodichloromethane	0.111	0.352	Propylene	0.110	0.351
Bromoform	0.183	0.583	Styrene	0.155	0.493
Bromomethane	0.0448	0.143	*tert*-Amyl Methyl Ether	0.0518	0.165
Carbon Disulfide	0.239	0.762	Tetrachloroethylene*	0.0992	0.315
Carbon Tetrachloride*	0.0840	0.267	Toluene	0.493	1.57
Chlorobenzene	0.0887	0.282	*trans*-1,2-Dichloroethylene	0.0533	0.169
Chloroethane	0.0659	0.209	*trans*-1,3-Dichloropropene	0.0807	0.257
Chloroform*	0.0633	0.201	Trichloroethylene*	0.0806	0.256
Chloromethane	0.0961	0.306	Trichlorofluoromethane	0.0654	0.208
Chloroprene	0.0469	0.149	Trichlorotrifluoroethane	0.0749	0.238
cis-1,2-Dichloroethylene	0.0740	0.235	Vinyl Chloride*	0.0327	0.104

*NATTS Tier I compounds

Table 11-13. 2018 Carbonyl Method Detection Limits (Underivatized Concentration)

Compound	MDL ($\mu g/m^3$)	SQL ($\mu g/m^3$)
2,5-Dimethylbenzaldehyde	0.0163	0.05171
2-Butanone (Methyl Ethyl Ketone)	0.136	0.432
Acetaldehyde *	0.0389	0.124
Acetone	0.408	1.30
Benzaldehyde	0.00952	0.03029
Butyraldehyde	0.0576	0.183
Crotonaldehyde	0.00809	0.02571
Formaldehyde *	0.0739	0.235
Hexaldehyde	0.00742	0.02361
Isovaleraldehyde	0.0112	0.03565
Propionaldehyde	0.00469	0.01493
Tolualdehydes	0.0169	0.05361
Valeraldehyde	0.00746	0.02372

NOTE: Assumes 1000 L sample volume. MDLs determined in June 2018.
*NATTS Tier I compounds

Table 11-14. 2018 PAH Method Detection Limits

Compounds	MDL (ng/m^3)	SQL (ng/m^3)
9-Fluorenone	0.0607	0.193
Acenaphthene	0.0743	0.236
Acenaphthylene	0.0147	0.0466
Anthracene	0.0134	0.0426
Benzo(a)anthracene	0.0104	0.0330
Benzo(a)pyrene *	0.0106	0.0337
Benzo(b)fluoranthene	0.0213	0.0677
Benzo(e)pyrene	0.0105	0.0334
Benzo(g,h,i)perylene	0.0130	0.0413
Benzo(k)fluoranthene	0.0116	0.0369
Chrysene	0.00805	0.0256
Coronene	0.00467	0.0148
Cyclopenta(c,d)pyrene	0.00711	0.0226
Dibenz(a,h)anthracene	0.0150	0.0477
Fluoranthene	0.0248	0.0790
Fluorene	0.0693	0.220
Indeno(1,2,3-cd)pyrene	0.0133	0.0424
Naphthalene *	1.82	5.77
Perylene	0.00929	0.0295
Phenanthrene	0.125	0.398
Pyrene	0.0126	0.0400
Retene	0.0617	0.196

NOTE: Assumes a 300 m^3 sample volume. MDLs determined in May 2018.
*NATTS Tier I compounds

Two MDLs are determined for the metals analysis. One is determined for quartz filters, and the other for Teflon filters. The detection limits for metals the determined by the FAC[(20)] method using compiled method blank data. If the resulting MDL for any element does not meet criteria, then seven to 10 replicate blank filter strips should be spiked at a concentration of two to five times the estimated MDL, digested, and analyzed to determine the MDL values using the method described in 40 CFR Part 136[(18)], Appendix B. Both procedures should be prepared following the entire analytical method procedure. The metals MDLs are shown in Table 11-15 and are based on a sampling volume of 2000 m^3 for the quartz filters and 24.04 m^3 for the Teflon filters. For 2018, the FACA procedure was used to determine the MDLs for the quartz and Teflon filters. The hexavalent chromium MDL is also included in Table 11-15 and is based on a sampling volume of 21.6 m^3.

The Sample Quantitation Limit (SQL) is also reported in Table 11-13 through Table 11-15. The SQL is defined as the lowest concentration an analyte can be reliably measured within specified limits of precision and bias during routine laboratory operating conditions. The SQL is defined by EPA as a multiplier (3.18) of the MDL and is considered the lowest concentration that can be accurately measured, as opposed to just detected. ERG submits this data into AQS using flags to show where the data is in respect to the detection level.

The NATTS Program requires sampling and analysis for 18 target air toxic analytes. Hexavalent chromium is no longer required by the NATTS program, but was given a target MDL in the latest NATTS TAD(18) and the NATTS Work Plan Template(21). The NATTS program uses sensitivity to assess quantification from a monitoring site with the appropriate level of certainty. In order to meet this objective, target MDLs have been established for the NATTS Program and are compared to the current 2018 ERG MDLs in Table 11-16.

Table 11-15. 2018 Metals Method Detection Limit

Element	47 mm Teflon MDL (ng/m^3)	47 mm Teflon SQL (ng/m^3)	8x10" Quartz MDL (ng/m^3)	8x10" Quartz SQL (ng/m^3)
Antimony *	0.151	0.479	0.0336	0.107
Arsenic *	0.0362	0.115	0.00879	0.0280
Beryllium *	0.00142	0.00453	0.00130	0.00414
Cadmium *	0.00487	0.0155	0.00544	0.0173
Chromium *	3.27	10.4	1.13	3.61
Cobalt *	0.0842	0.268	0.0183	0.0582
Lead *	0.0657	0.209	0.0855	0.272
Manganese *	0.194	0.616	0.816	2.60
Mercury	0.0153	0.0485	0.00498	0.0158
Nickel *	1.21	3.85	0.436	1.39
Selenium *	0.0582	0.185	0.0101	0.0321
Hexavalent Chromium MDL (47mm Cellulose)				
Hexavalent Chromium	0.0040	0.0127		

NOTE: For total metals: Assumes total volume of 24.04 m^3 for Teflon filters and 2000 m^3 for Quartz filters.
For hexavalent chromium: Assumes total volume of 21.6 m^3.

*NATTS Tier I Compounds

Table 11-16. Target MDLs for the NATTS Program

Pollutant	NATTS Target MDL (µg/m^3)	ERG 2018 MDL (µg/m^3)	Is ERG MDL < Target MDL?		
NATTS Tier I VOC HAPs					
Acrolein	0.09	0.516	NO		
Benzene	0.13	0.0463	YES		
1,3-Butadiene	0.10	0.0429	YES		
Carbon Tetrachloride	0.17	0.0840	YES		
Chloroform	0.50	0.0633	YES		
Tetrachloroethylene	0.17	0.0992	YES		
Trichloroethylene	0.20	0.0806	YES		
Vinyl Chloride	0.11	0.0327	YES		
NATTS Tier I Carbonyl HAPs					
Acetaldehyde	0.45	0.0389	YES		
Formaldehyde	0.080	0.0739	YES		
Pollutant	**NATTS Target MDL (ng/m^3)**	**ERG 2018 MDL (ng/m^3)**	**Is ERG MDL < Target MDL?**		
NATTS Tier I PAH HAPs					
Benzo(a)pyrene	0.91	0.0106	YES		
Naphthalene	29	1.82	YES		
NATTS Tier I Metal HAPs					
		(Low Vol PM_{10})		*(High Vol PM_{10})*	
Arsenic (PM_{10})	0.23	0.0362	YES	0.00879	YES
Beryllium (PM_{10})	0.42	0.00142	YES	0.00130	YES
Cadmium (PM_{10})	0.56	0.00487	YES	0.00544	YES
Lead (PM_{10})	15.0	0.0657	YES	0.0855	YES
Manganese (PM_{10})	5.0	0.194	YES	0.816	YES
Nickel (PM_{10})	2.1	1.21	YES	0.436	YES

NOTE: Target MDL's were obtained from the NATTS Work Plan Template (March 2015), Section 3.1 and the NATTS TAD, Revision 3[18]

SECTION 12
INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS

To ensure the quality of the sampling and analytical equipment, ERG conducts performance checks for all equipment used in each of the programs. ERG checks the sampling systems annually, and makes repairs as needed. ERG tracks the performance of the analytical instrumentation to ensure proper operation. ERG also maintains a spare parts inventory to shorten equipment downtime. Table 12-1 details the maintenance items, how frequently they will be performed, and who is responsible for performing the maintenance. All checks, testing, inspections, and maintenance done on each instrument are recorded in the appropriate Maintenance Logbook or LIMS Instrument Maintenance Logs for each instrument.

Table 12-1
Preventive Maintenance in ERG Laboratories

Item	Maintenance Frequency	Responsible Party
For Analytical Systems		
Multipoint Calibration	As needed or at least at intervals specified in Section 11	Analyst
Comparison to Continuing Calibration Standard	Daily	Analyst
Replace GC/LC/IC Column	As necessary (i.e., observe peaks tailing, retention time shifts, increased baseline noise, etc.)	Analyst
Detector Maintenance	As necessary	Analyst
Computer Backup	Biweekly, Daily preferred	Analyst
Accelerated Solvent Extractor		
Piston Rinse Seal	Quarterly, or as needed	Analyst
Standard Rinse Seal	Quarterly, or as needed	Analyst

Table 12-1
Preventive Maintenance in ERG Laboratories (Continued)

Item	Maintenance Frequency	Responsible Party
High Performance Liquid Chromatography		
In-line filter	As necessary (when pressure increases above 2500 psi)	Analyst
Inspect Delivery System Motor	Annually	Service Technician
Replace Teflon Delivery Tubing	Annually	Service Technician
Ion Chromatography		
Rinse Post Column Reagent lines with methanol	As necessary	Analyst
Rinse Eluent Lines with Deionized water	After every sequence	Analyst
Sonicate Inlet and Outlet Check Valves	As necessary	Analyst
Rinse Autosampler Injector	As necessary	Analyst
Inorganic Laboratory		
Flush system for 5 minutes with the plasma on with a rinse blank	After every sequence	Analyst
Cleaning cones, torch, injector, spray chamber	Quarterly, or as needed for analysis quality	Analyst
Change Roughing Pump Oil	Annually	Service Engineer
Replace Air Filters	Annually	Service Engineer
For Sampling Field Equipment (UATMP, Carbonyl, NMOC/SNMOC, and Hexavalent Chromium)		
Inspect/Replace vacuum pump diaphragms and flapper valves	At each system certification effort	ERG
Inspect Sampler (overall)	At each system certification effort and prior to each scheduled collection event	ERG/Field Operator
Inspect/Replace Cartridge Connectors	Prior to each collection event, replace as needed	ERG/Field Operator
Replace Ozone Scrubber	At each system certification effort	ERG
MFM Check or Flow check	At each system certification effort	ERG
Inspect/Replace Fans	At each system certification effort	ERG

12.1 SNMOC, VOC, and PAMS

The GC/FID/MS systems are maintained under a service agreement. ERG personnel perform minor maintenance, such as filament changes, carrier gas filter replacements, column maintenance, and source cleaning. The following spare parts should be kept in the lab: traps, filament, column, and split for the column. All procedures, checks, and scheduled maintenance checks for VOC GC/FID/MS analysis are provided in ERG's SOP (ERG-MOR-005) presented in Appendix C.

12.2 Carbonyls

The carbonyl HPLC analytical systems are maintained under a service agreement. ERG personnel perform minor maintenance, such as column and detector maintenance, on an as-needed basis. The following spare parts should be kept in the lab: solvent frit, column, in-line filter and guard column. All procedures, checks, and scheduled maintenance checks are provided for carbonyl HPLC analysis in ERG's SOP (ERG-MOR-024) presented in Appendix C.

12.3 HAPs

The GC/MS systems for PAH and VOC analysis are maintained under the same service agreement. ERG personnel perform minor maintenance as needed. The following spare parts should be kept in the lab: injector sleeve, filament, and column.

For the HAPs sample analyses performed on the ICP-MS and IC, routine preventive maintenance is performed by the Analyst or Task Lead. ERG personnel perform minor maintenance, such as column and detector maintenance, on an as-needed basis. Contracted service agreements are in place for non-routine maintenance. Spare pump tubing, focusing lens, gem tips, and o-rings should be kept in the lab for the ICP-MS. A spare guard and analytical column, piston seals, reaction coil, and reservoir frits should be kept in the lab for the IC. More procedures, checks, and scheduled maintenance checks are provided in ERG's SOP

(ERG-MOR-049) for PAH analysis by GC/MS, ERG-MOR-095 for metals analysis by ICP-MS, and ERG-MOR-063 for hexavalent chromium by IC presented in Appendix C.

SECTION 13
INSTRUMENT CALIBRATION AND FREQUENCY

The programs are discussed separately in this section because the requirements for analytical system calibrations differ. Analytical instruments and equipment are calibrated when the analysis is set up, when the laboratory takes corrective action, following major instrument maintenance, or if the continuing calibration acceptance criteria have not been met. Appropriate standards are prepared by serial dilutions of pure substances or accurately prepared concentrated solutions. Many analytical instruments have high sensitivity, so calibration standards must be extremely dilute solutions. In preparing stock solutions of calibration standards, great care is exercised in measuring weights and volumes, since analyses following the calibration are based on the accuracy of the calibration.

Each calibration analysis is stored, electronically and hardcopy, with traceability for the samples analyzed using that calibration. Each of the analytical systems is calibrated for all reported target analytes, except for the NMOC and SNMOC calibrations. The NMOC calibration is based on propane and the SNMOC calibration is based on propane, hexane, benzene, octane, and decane average response factors. NMOC calibration will be discussed in more detail when the analysis is requested by a State.

13.1 SNMOC Calibration

For the SNMOC method, average carbon response factors are obtained quarterly (at a minimum) based on the analysis of humidified calibration standards prepared in canisters. The Dynamic Flow Dilution System (SOP Number ERG-MOR-061, Appendix C) is used to dilute certified Linde or equivalent alkanes into clean, evacuated SUMMA®- treated canisters. The gas standards are traceable via the gravimetric preparation using NIST-traceable weights. These gas standards are recertified annually. HPLC grade water is used to humidify the standard to approximately 50 percent. The standard is diluted with scientific-grade air to achieve the desired concentrations for the calibration. The response factors generated from the calibration are used to determine concentrations of detected compounds, on the assumption that FID response is linear with respect to the number of carbon atoms present in the compound.

At least five calibration standards are prepared in ranges from 5 to 400 ppbC concentrations. The average response factors for propane, hexane, benzene, octane, and decane are determined using the response correlated to concentration. Individual concentrations for the C_2 through C_{13} compounds detected on the FID are calculated using one of the five response factors, with a similar Carbon number. The calibration is considered representative if the average RF RSD for the curve is within ±20 percent. Daily, before sample analysis, a CCV standard (such as Air Environmental gas standard), is analyzed to ensure the validity of the current response factors. Ten selected hydrocarbons, ranging from C_2 through C_{10}, from the QC standard are compared to the calculated theoretical concentrations. A percent recovery of 70-130 percent is considered acceptable showing the analytical system is in control.

A blank of cleaned, humidified air or N_2 is analyzed after the CCV and before sample analyses. The system is considered in control if the total NMOC concentration for the blank is less than or equal to 20 ppbC.

13.2 VOC Calibration

Calibration of the GC/FID/MS is accomplished quarterly (at a minimum) by analyzing humidified calibration standards prepared in canisters generated from NIST-traceable Linde or Air Environmental (or equivalent) gas standards. The certified standards contain the VOC target compounds at approximately 500 ppbV. Although the MS is the primary quantitation tool, responses on the FID are recorded to detect and quantify hydrocarbon peaks and can be used for SNMOC or PAMS results. The calibration for these hydrocarbon peaks should be accomplished as explained in Section 13.1.

Calibration standards are prepared with a dynamic flow dilution apparatus (Figure 13-1, see Standard Operating Procedure ERG-MOR-061, Appendix C). The gases are mixed in a SUMMA®-treated mixing sphere and bled into evacuated canisters. One dilution air stream is humidified by routing it through a SUMMA®- treated bubbler containing HPLC-grade water; the other stream is not humidified. The dilution air streams are then brought together for mixing with the streams from the certified cylinders. Flow rates from all streams are gauged and controlled by mass flow controllers. The split air dilution streams are metered by "wet" and "dry" rotameters (~50 percent relative humidity) from the humidified and unhumidified dilution air streams, respectively.

The system is evacuated with a vacuum pump while the closed canister is connected. The lines leading to the canister and to the mixing sphere are flushed for at least 20 minutes with standard gas before being connected to the canister for filling. A precision pressure gauge measures the canister pressure before and after filling.

Initial calibration standards are prepared at nominal concentrations of 0.25, 0.5, 1, 2.5, 5, and 10 ppbV for each of the target compounds (a minimum of 5 levels are required). All standards and samples are analyzed with the following internal standards: *n*-hexane-d_{14}, 1,4-difluorobenzene, and chlorobenzene-d_5. The calibration requires average response factors, based on the internal standard, of ± 30 percent RSD, however per Compendium Method TO-15[4] acceptance criteria, up to two compounds can have ± 40 percent RSD (non-Tier I compounds). The CCV is made from a second source certified gas at an average concentration of 2.5 ppbV. The CCV must have RRFs within ± 30% of the mean initial calibration RRFs.

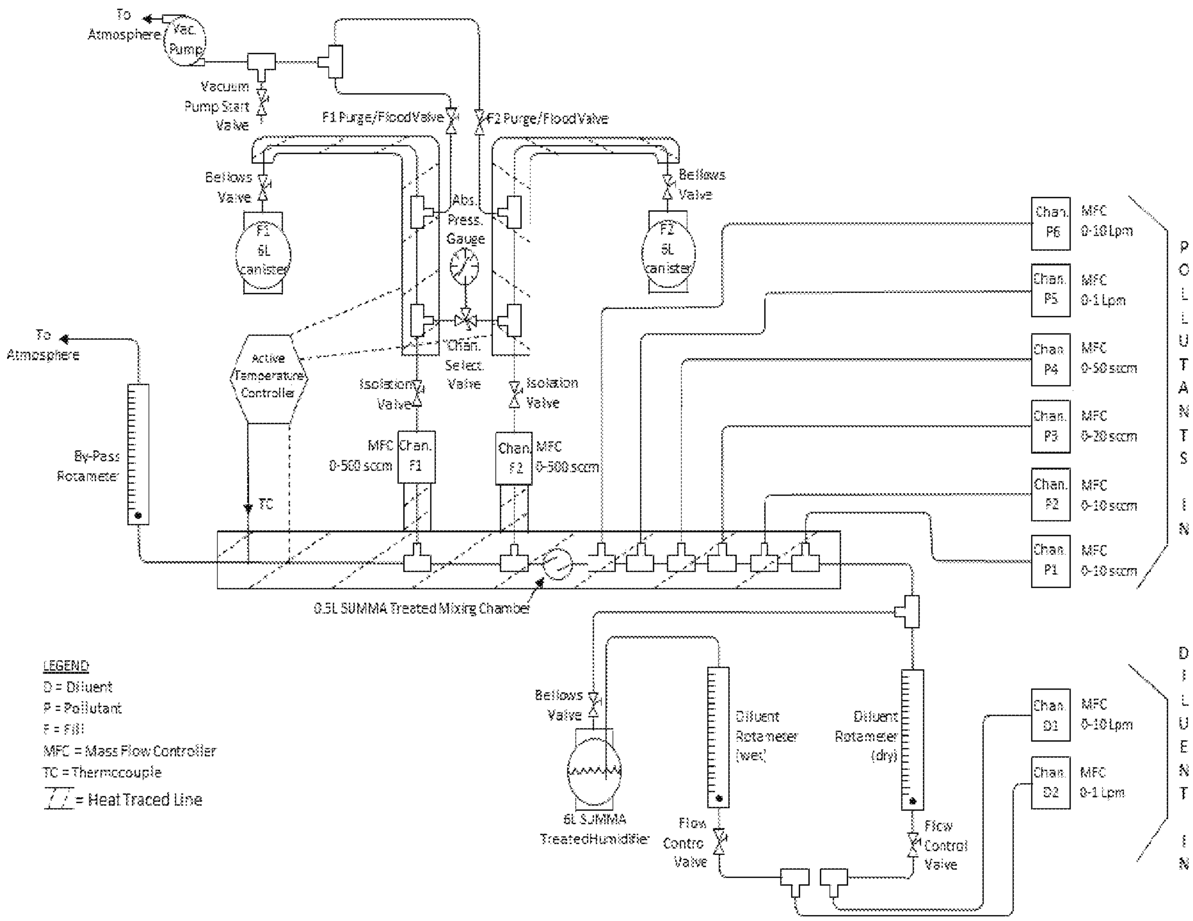

Figure 13-1. Dynamic Flow Dilution Apparatus

13.3 Carbonyl Calibration

For the carbonyl analyses, the HPLC instrument is calibrated using an acetonitrile solution containing the derivatized targeted compounds. The calibration curve consists of six concentration levels ranging from 0.01 to 3.0 microgram per milliliter (μg/mL) (underivatized concentration), and each is analyzed in triplicate. The standard linear regression analysis performed on the data for each analyte must have a correlation coefficient greater than or equal to 0.999. The Relative Error (RE) for each compound at each level against the calibration curve must be ≤ 20 percent. As a QC procedure to verify the calibration and check HPLC column efficiency, a SSQC sample solution containing target carbonyl compounds at a known concentration is analyzed in triplicate after every calibration curve, with an 85-115 percent recovery criterion.

In each sequence, a CCV (a second source standard) is analyzed every 12 hours or less while samples are analyzed (meeting the 85-115 percent recovery criterion). A system blank brackets the analytical batch, by analyzing one blank at the beginning and one at the end of each sequence.

13.4 HAPs Calibration

The GC/MS system in SIM mode is calibrated for PAH analysis at a minimum every six week. The average calibration RRF must be greater than or equal to the minimum RRF presented in Table 13-1. For the other HAPs sample analyses, calibration is performed on the ICP-MS and IC. Calibration requirements for the HAPs analytical methods are in Tables 11-7, 11-9 and 11-10.

Table 13-1.
Relative Response Factor Criteria for Initial Calibration of Common Semivolatile Compounds

Semivolatile Compounds	Minimum RRF	Maximum %RSD	Maximum % Difference
Naphthalene	0.700	30	30
Acenaphthylene	1.300	30	30
Acenaphthene	0.800	30	30
Fluorene	0.900	30	30
Phenanthrene	0.700	30	30
Anthracene	0.700	30	30
Fluoranthene	0.600	30	30
Pyrene	0.600	30	30
Benz(a)anthracene	0.800	30	30
Chrysene	0.700	30	30
Benzo(b)fluoranthene	0.700	30	30

Note – The ASTM method includes no minimum RRF criteria, therefore none are listed here for the ASTM[12] compounds.

Table 13-1.
Relative Response Factor Criteria for Initial Calibration of Common Semivolatile Compounds (Continued)

Semivolatile Compounds	Minimum RRF	Maximum %RSD	Maximum % Difference
Benzo(k)fluoranthene	0.700	30	30
Benzo(a)pyrene	0.700	30	30
Indeno(1,2,3-cd)pyrene	0.500	30	30
Dibenz(a,h)anthracene	0.400	30	30
Benzo(g,h,i)perylene	0.500	30	30
Perylene	0.500	30	30
Coronene	0.700	30	30
Benzo(e)pyrene	--	30	30
Cyclopenta(c,d)pyrene	--	30	30
Retene	--	30	30
9-Fluorenone	--	30	30

Note – The ASTM method includes no minimum RRF criteria, therefore none are listed here for the ASTM[12] compounds.

13.5 Laboratory Support Equipment Calibration

Analytical balances are serviced and calibrated annually with NIST traceable weights by a vendor service technician. The certificate of Weight Verification (ISO9001) is kept on file by the QA Coordinator. The balance calibrations are checked daily on days of use with Class 1 weights and recorded. The data loggers used for temperature/humidity/pressure have calibration checks annually performed by the vendor. The infrared (IR) thermometers are annually vendor calibrated with NIST-traceable standards. The calibration of the thermometers used in the metals sample digestion procedure are checked against a thermometer with a NIST traceable vendor calibration. The pressure gauges used for measuring sample canister pressure at receipt are calibrated annually by a certified vendor. Other pressure gauges, used in canister cleaning or canister sample dilution, are checked against a “transfer standard” gauge that is calibrated annually by a certified vendor. MFCs used in the canister dynamic dilution standard system are calibrated annually and the calibrations are checked quarterly.

Pipette calibrations are checked and recorded quarterly. If a pipette fails a calibration check they are rechecked. If it continues to fail, it is sent back to the manufacturer for recalibration. If recalibration is not possible it will be repaired or replaced with a new pipette. Syringe calibrations are checked and recorded annually. If a syringe fails the calibration check, it will be replaced with a new one. Class A volumetric glassware is used throughout the laboratory for bringing sample extracts up to final volume.

SECTION 14

INSPECTION/ACCEPTANCE FOR SUPPLIES AND CONSUMABLES

14.1 Purpose

The purpose of this element is to establish and document a system for inspecting and accepting all supplies and consumables that may directly or indirectly affect the quality of the NMP. By having documented inspection and acceptance criteria, consistency of the supplies can be assured. This section details the supplies/consumables, their acceptance criteria, and the required documentation for tracing this process.

14.2 Critical Supplies and Consumables

Table 14-1 details the various components for the field and laboratory operations.

14.3 Acceptance Criteria

Acceptance criteria for supplies/consumables must be consistent with overall project technical and quality criteria. As requirements change, so do the acceptance criteria. Knowledge of laboratory equipment and experience are the best guides to acceptance criteria. It is the laboratory analyst's responsibility to update the criteria for acceptance of consumables. Other acceptance criteria such as observation of damage due to shipping can only be performed once the equipment has arrived on site.

All supplies and consumables are inspected and accepted or rejected upon receipt in the laboratory. The ERG employee who ordered the supply is responsible for verifying that the order is acceptably delivered, stored and dispersed upon receipt in the laboratory. The recipient's signature on the packing slip indicates the received goods were received and are acceptable. Some supplies or consumables listed in Table 14-1 must be deemed acceptable through testing or blanking, such as with the carbonyl DNPH cartridges. Any changes in standards and sample media must meet the acceptance criteria outlined in Section 11 for that particular method. Such testing and blanking data is kept with the sample data. Staff should not use supplies or consumables of different model numbers or grades without first discussing it with the Program Manager and specific Task Leader and testing the supply or consumable. Staff should keep any certificate of analysis or cleanliness that arrives with the supply/consumable on file. For specific information on reagents and standards used, see applicable method SOP.

Table 14-1
Critical Supplies and Consumables

Area	Item	Description	Vendor	Model Number
Field Supplies and Consumables (Fabrication Lab)				
All Samplers	Various Swagelok® fittings	All Samplers	Swagelok	Various
NMOC Sampler	Pump	Metal Bellows	KNF Newberger	UN 05-SV.91
VOC Sampler	Vacuum Pump	VOC System	Thomas	2107VA20
	Canisters	VOC Canisters	Entech	6-liter Silonite® Canisters
Carbonyl Sampler	DNPH Cartridges	DNPH coated plastic cartridges	Waters	WAT 037500
Hexavalent Chromium Sampler	Pump	High Vacuum	Thomas	VA-2110
Laboratory Supplies and Consumables (Laboratories listed below)				
All Laboratories	Powder Free Gloves	Polyethylene	VWR	32915-246
All Laboratories	Gloves	Nitrile	Expotech,ThermoFisher, VWR	1461558 (Expotech)
Liquid Chromatography	Guard column	Zorbax ODS	Agilent	820950-902
Liquid Chromatography	Chromatographic Column	Zorbax ODS	Agilent	880952-702
Liquid Chromatography	UV Lamp	For 2487 detector	Waters	WA 5081142
GC/MS – VOC	Chromatographic Column	0.32 x 1 µ - 60 m column	Restek	Rxi-lms
GC/MS – SVOC	Chromatographic Column	0.25 x 0.25 µ - 30 m column	Agilent J&W	HP-5MS UI
GC/MS – SVOC	Inject seal	Injection port seal	Expotech	2264837
GC/MS – SVOC	Liner	Injection port liner	Expotech	2377232

Table 14-1
Critical Supplies and Consumables (Continued)

Area	Item	Description	Vendor	Model Number
GC/MS & Liquid Chromatography	Helium	Carrier Gas	Air Gas	UHP
GC/MS	Hydrogen Gas	FID Gas	Air Gas	UHP
GC/MS	Liquid Nitrogen	Coolant Gas	Air Gas	Bulk
GC/MS	Liquid Argon	Coolant Gas	Air Gas	Bulk
GC/MS	Air	FID Gas	Air Gas	Zero
GC/MS	Traps	Glass bead/Tenax Trap	Entech	01-04-11340
GC/MS	Trap Heater	Sample Trap Heater	Entech	01-09-13010
GC/MS	Cryogenic Valve	Cryogenic Valve	Entech	01-01-71760
ICP-MS	Liquid Argon	Coolant Gas	Air Gas	Bulk
ICP-MS	Acid	High Purity Nitric	Fisher/SCP Science	A200-212/Plasma Pure Plus
ICP-MS	Acid	Hydrochloric Acid	Fisher/SCP Science	A466-1/Plasma Pure Plus
ICP-MS	Hydrogen Peroxide	Hydrogen Peroxide, 30%	SCP Science	Plasma Pure Plus
ICP-MS	Whatman 8"x11" Quartz/Glass Fiber Filters MTL 47mm Teflon™ Filters	Filters	GE Healthcare Life Sciences & MTL	1851-8531 1882-8532 PT47-EP
IC	Reaction Coil	Knitted Reaction Coil	ThermoFisher	042631
IC	Guard Column	Dionex Ion Pac NG1	ThermoFisher	039567
IC	Analytical Column	Dionex Ion Pac AS7	ThermoFisher	035393
IC	Methanol	Solvent	Expotech, Fisher, VWR	HPLC grade
IC	Sample vials 14 mL, polystyrene with caps	Sample containers	ThermoFisher	352057
IC	Whatman Filters	Filters–47mm ashless cellulose	Expotech, Fisher	09-850H
Prep	Water Filter	Ultrapure Ion Exchange Cartridge	Expotech	1425973
Prep	Water Filter	Cartridge submicron	Expotech	1425977
Prep	Water Filter	Pretreatment Cartridge	Expotech	1426051
Prep	Whatman Filters	Filters–110mm GFA	Expotech	1422153

Table 14-1
Critical Supplies and Consumables (Continued)

Area	Item	Description	Vendor	Model Number
Prep	PUF	Pre-cleaned PUF	Cen-Med, Expotech	824-20038, 2256468
Prep	XAD®	XAD®	Expotech	2255045
Prep	Petri Dish	Filter container	Expotech	1426833
Prep	Acetonitrile	Solvent	Expotech, Fisher, VWR	HPLC grade
Prep	Methylene Chloride	Solvent	Expotech, Fisher, VWR	Optima grade
Prep	Hexane	Solvent	Expotech, Fisher, VWR	95% (Optima grade)
Prep	Toluene	Solvent	Expotech, Fisher, VWR	Optima Grade
Prep	Nitrogen	Evaporation gas	Air Gas	UHP (or Bulk)
Prep	Amber glass bottles 250 mL	Sample containers	Expotech	2373176
Prep	Extraction cells	Sample containers	Thermo Electron	068077
Prep	Ottawa sand	Extraction filler	Expotech	2262138
Prep	Seals	ASE Vespel Seals	Fisher	056776
Prep	Disposable pipets	Disposable pipets	Expotech	1405717
Prep	4 mL amber sample vials	Sample containers	Expotech, Fisher, VWR	66030-734 (VWR)
Prep	4 mL sample Teflon lined caps	Sample containers	Expotech, Fisher, VWR	66030-771 (VWR)
Prep	Autosampler snap-it vials	Sample containers	Waters	WAT 094220
Prep	Autosampler snap-it caps	Sample containers	Waters	18000303

Consumables and supplies with special handling and storage needs must be handled and stored as suggested by the manufacturer. Consumables with expiration dates, such as solvents and standards, must be labeled with a receipt date, date opened, and the initials of the person that opened the consumable and standard expiration dates must be entered into the standards section of LIMS. To decrease waste, the oldest supplies or consumables should be used first.

SECTION 15
DATA MANAGEMENT

15.1 Data Recording

Data management for sample data is presented in Figure 15-1. The sample data path is shown from sample origination to data reporting and storage. The LIMS allows the laboratory to manage and track samples, instrument workflow, and reporting. The LIMS stores the raw instrument data and performs the conversion calculations to put the data into final reporting units. These calculations are reviewed and documented annually by the QA coordinator and kept in the QA files in Room 102. The main procedures are described in the *SOP for the Laboratory Information Management System* (ERG-MOR-099). The main functions of the LIMS system include, but are not limited to:

- Sample login;
- Sample scheduling, and tracking;
- Sample processing and quality control; and
- Sample reporting and data storage.

All LIMS users must be authorized by the LIMS Administrator and permitted specified privileges. The following privilege levels are defined:

- Data Entry Privilege – The individual may see and modify only data within the LIMS that he or she has personally entered.
- Reporting Privilege – Without additional privileges.
- Data Administration Privilege – Data Administrators for the database are allowed to change data as a result of QA screening and related reasons. Data Administrators are responsible for performing the following tasks on a regular basis:
  - Merging/correcting the duplicate data entry files;
  - Running verification/validation routines, correcting data as necessary.

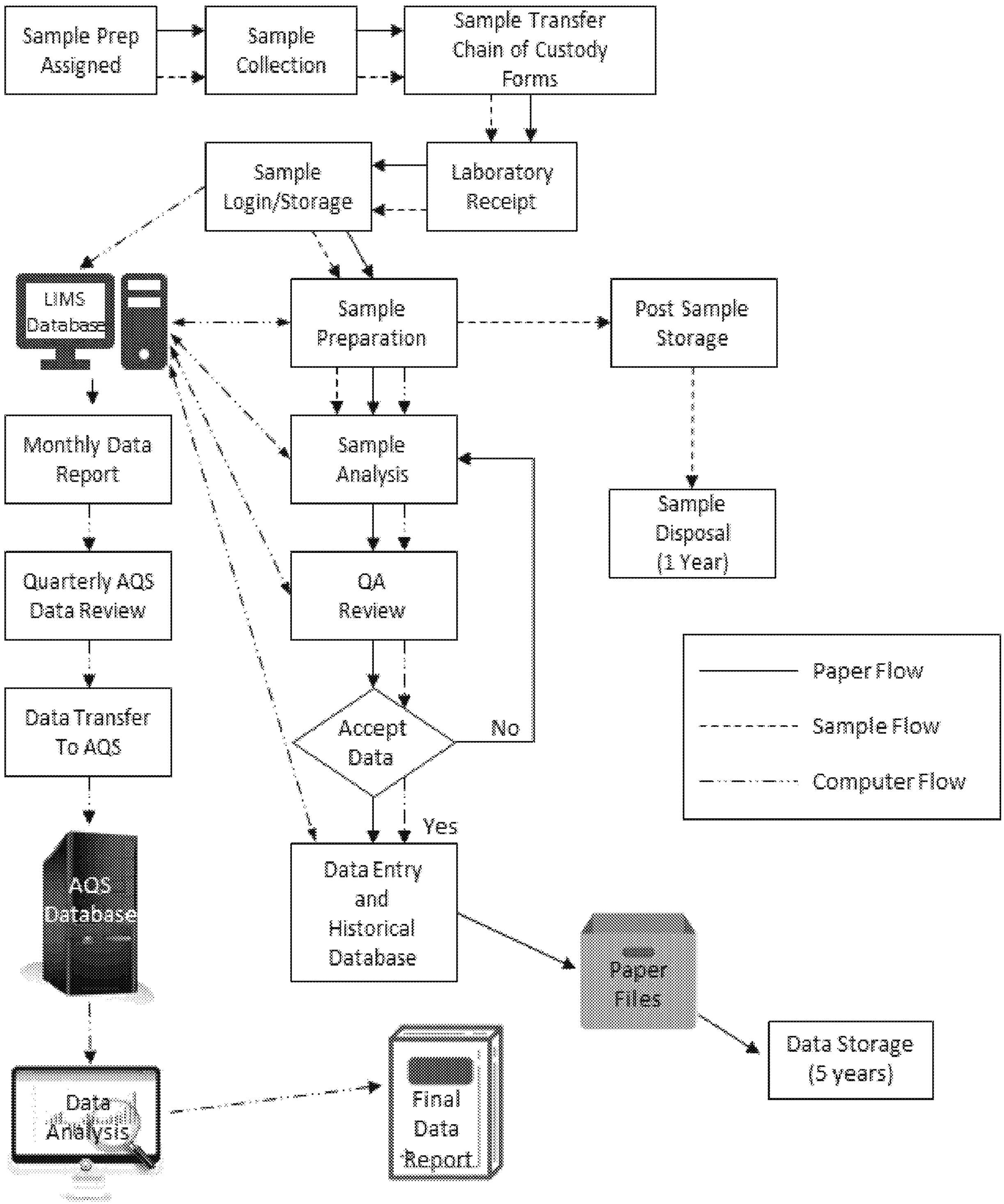

Figure 15-1. Data Management and Sample Flow Diagram

15.2 Data Validation

Data validation is a combination of checking that data processing operations have been carried out correctly and of monitoring the quality of the field operations. Data validation is confirmed by examination of objective evidence that the requirements for a specific intended use are fulfilled as presented in Section 4. This data validation is performed prior to the annual final report. The data reported monthly are considered preliminary until the data is validated, entered into the AQS database, and reported in the annual final report. Data validation is discussed in more detail in Section 18.5.

15.3 Data Reduction and Transformation

Data generated on an instrument is reduced by the analyst via instrument chromatographic software. Any manual integration to chromatographic data follows SOP ERG-MOR-097, the *SOP for Manual Integration of Chromatographic Peaks*. Specific equations used by the instrument chromatographic software to calculate concentration are documented in the individual analytical SOPs found in Appendix C. The equations for transforming raw data are set up to automatically calculate to final concentrations in the LIMS system. The initial and final reporting units for SNMOC are ppbC. All other analyses are reported in units different from their raw data. The initial units for the Carbonyl Compounds analysis are microgram per milliliter (μg/mL), while the final reporting units are in either ppbV or microgram per cubic meter ($\mu g/m^3$), per site request, however the NATTS sites are to be reported in $\mu g/m^3$ per the NATTS TAD(18). The initial units for VOC are ppbV and the LIMS data reports are in ppbV and $\mu g/m^3$. The PAH initials units are ng/μL with final reporting units of nanogram per cubic meter (ng/m^3). The initial units for metals are ng/L with final reporting units of ng/m^3. The initial units for the hexavalent chromium analysis are ng/mL with final reporting units of ng/m^3. The associated MDLs are reported in final reporting units with the final concentrations. MDLs are adjusted for dilution and actual prep volumes, and sample collection volume where applicable, before reporting.

The electronic data file is uploaded onto a network server (which is backed-up daily) and into the LIMS. Once the data is in LIMS, the Task Leader reviews it following the checklists presented in the SOPs using instrument software and the method-specific control limits set up in LIMS. Ten percent of all data is reviewed by the QA Coordinator or designee following the checklist and method specific acceptance criteria in the summary quality control procedure tables outlined in Section 11. After data has successfully completed both reviews and the checklists have been signed, it is available for reporting by the Program Manager.

The *SOP for Project Peer Review* uses manual calculations and visual verification to review all data reported to EPA and State/Local/Tribal agencies following guidelines outlined in SOP ERG-MOR-057 (see Appendix C). *SOP for Developing, Documenting, and Evaluating the Accuracy of Spreadsheet Data*, presented in SOP ERG-MOR-017 (see Appendix C), is consulted in special cases where the calculations are performed via spreadsheets instead of the LIMS system.

Reporting formats are designed to fulfill the program requirements and to provide comprehensive, conventional tables of data. The LIMS data reporting format includes any required data qualifiers, footnotes, detection limits for each analyte, and appropriate units for all measurements. The LIMS can produce Adobe and Excel data reports, which is standard for this program. Each report is reviewed by the Program Manager or designee before it is sent to the client.

15.4 Data Transmittal

Data transmittal occurs when data are transferred from one person or location to another or when data are copied from one form to another. Some examples of data transmittal are copying raw data from a notebook onto a data entry form for keying into a computer file and electronic transfer of data over a computer network. Each individual SOP listed in Appendix C discusses the procedures for determining the calculations of concentrations as well as data entry.

ERG will report all ambient air quality data and information specified by the AQS User's Guide and other documents located at the website http://www.epa.gov/ttn/airs/airsaqs/manuals/ coded in the AQS format. Such air quality data and information will be fully screened and validated and will be submitted directly to the AQS database via electronic transmission, in the format of the AQS, and in accordance with the annual schedule. The *SOP for the Preparation of Monitoring Data for AQS Upload* is presented in Appendix C (SOP ERG-MOR-098).

15.5 Data Summary

ERG is implementing the data summary and analysis program in the form of a final annual report. The following specific summary statistics will be tracked and reported for the network:

- Single sampler bias or accuracy (based on laboratory audits if available);
- Analytical precision (based on analytical replicates);
- Sampler precision (based on collocated data);
- Network-wide bias and precision; and
- Data completeness.

Equations used for these reports are given in Table 15-1.

Table 15-1. Report Equations

Criterion	Equation
Coefficient of Variation (CV)- *p* and *r* are concentrations from the primary and duplicate samplers, respectively. This equation is also used for collocated samples and replicate analysis.	$CV = 100 \times \sqrt{\frac{\sum_{i=1}^{n}\left[\frac{(p-r)}{0.5 \times (p+r)}\right]^2}{2n}}$
Percent Completeness	$\text{Completeness} = \frac{N_{\text{valid}}}{N_{\text{theoretical}}} * 100$ Where, N_{valid} is the number of valid samples analyzed in the sampling year and $N_{\text{theoretical}}$ is the number of valid samples that should be taken within that same sampling year

15.6 Data Tracking

The ERG LIMS database contains the necessary input functions and reports appropriate to track and account for the status of specific samples and their data during processing operations. The following input locations are used to track sample and sample data status:

- Sample Control
  - Sample collection information (by Work Order);
  - Sample receipt/custody information;
  - Unique sample number (LIMS ID);
  - Storage location;
  - Required analyses;

- Laboratory
  - Batch/bench assignment;
  - Sequence assignment (if needed);
  - Data entry/review;
  - Query/update analysis status;
  - Standards/calibration information.

15.7 Data Storage and Retrieval

Data archival policies for hardcopy records are shown in Table 15-2.

All data are stored on the ERG LIMS server. This system has the following specifications:

- Operating System: Windows 2008 Server
- Memory: 6G RAM
- Hard Drives: Three drives of 450G each configured as RAID 5;
- Network card: Gigabit card (10/100/1000)
- Tape Drives for Backup: Two tape drives are daisy chained (HP StorageWorks, 1/8 G2 Tape Autoloader). Symantec Backup Exec Software ver. 12.5
- Security: Network login password protection on all workstations; Additional password protection applied by application software.

Security of the data in the database is ensured by the following controls:

- Password protection on the data base that defines three levels of access to the data;
- Logging of all incoming communication sessions, including the originating telephone number, the user's ID, and connect times; and
- Storage of media, including backup tapes, in an alternate location that is at a locked, restricted access area.

Table 15-2. Data Archive Policies

Data Type	Medium	Location	Retention Time	Final Disposition
Laboratory notebooks	Hardcopy	Laboratory	5 years after close of contract	N/A
LIMS Database	Electronic (on-line)	Laboratory	Backup media after 5 years	Backup tapes retained indefinitely

ASSESSMENT/OVERSIGHT

SECTION 16

ASSESSMENTS AND RESPONSE ACTIONS

An assessment is defined as an evaluation process used to measure the performance or effectiveness of the quality system or the establishment of the monitoring network and sites and various measurement phases of the data operation.

The results of QA assessments indicate whether the control efforts are adequate or need to be improved. Documentation of all QA and QC efforts implemented during the data collection, analysis, and reporting phases are important to data users, who can then consider the impact of these control efforts on the data quality. Both qualitative and quantitative assessments of the effectiveness of these control efforts will identify those areas most likely to impact the data quality. ERG will perform the following assessments to ensure the adequate performance of the quality system.

16.1 Assessment Activities and Project Planning

16.1.1 External Technical Systems and Data Quality Audits

A TSA is a thorough and systematic on-site qualitative audit, where facilities, equipment, personnel, training, procedures, subcontractor systems, and record keeping are examined for conformance to the QAPP. The TSAs will be performed by EPA or its designee at the ERG Laboratory. The TSAs for the contract are conducted approximately every 3 years. The EPA QA Office will implement the TSA either as a team or as an individual auditor. ERG will participate in any data quality audits by EPA or designee at the discretion of the EPA QA Coordinator.

The EPA audit team will prepare a brief written summary of findings for the Program Manager and Program QA Coordinator. Problems with specific areas will be discussed and an attempt made to rank them in order of their potential impact on data quality. ERG will work with

EPA to solve required corrective actions. As part of corrective action and follow-up, an audit finding response letter will be generated by the Program Manager and Program QA Coordinator. The audit finding response letter will address what actions are being implemented to correct the finding(s) of the TSA. This summary from EPA and the following response from ERG are filed in the QA/QC file in Room 102. The findings and the follow-up corrective actions are discussed in the annual QA Management Systems Review.

As part of ongoing National Environmental Laboratory Accreditation Conference (NELAC) certification, TSAs are performed at ERG by Florida Department of Health or designee every two years. A summary of findings is sent to ERG, specifically the QA Coordinator. The QA Coordinator sends its response of corrective actions which is either accepted or denied by Florida Department of Health. This documentation is stored in the QA/QC file in Room 102. The findings and the follow-up corrective actions are discussed in the annual QA Management Systems Review.

16.1.2 Internal Technical Systems Audits

An internal TSA is performed examining facilities, equipment, personnel, training, procedures, and record keeping for conformance to the individual SOPs and this QAPP. The TSAs will be performed by the Program QA Coordinator and will be conducted at least once per year. The checklists for the internal TSAs are based on the NATTS TSA or National Environmental Laboratory Accreditation Program (NELAP) checklists with additional areas addressing the individual SOPs and this QAPP. The content of the checklists vary episode to episode to ensure comprehensive in-depth coverage of procedures over time. Such elements will be included in the checklists:

- Criteria listed in Section 11 of this QAPP
- SOP specifications
- Method specifications
- Supporting equipment specifications
- Other laboratory wide QA systems in place (ex. Satellite SOP notebooks)

The Program QA Coordinator will report internal audit findings to the Program Manager within 30 days of completion of the internal audit in the form of a report. The EPA Delivery Order Manager will be informed if issues from the internal audit impact the quality of this program. The report is filed in the QA/QC file in Room 102. All corrective actions are addressed and implemented as soon as they are determined. The findings and the follow-up corrective actions are discussed in the annual QA Management Systems Review to assess effectiveness of the corrective actions.

16.1.3 Proficiency Testing

The PT is an assessment tool for the laboratory operations. 'Blind' samples are sent to the laboratory, where they follow the normal handling routines that any other sample follows. The results are sent to the Program Manager and Program QA Coordinator for final review and reporting to the auditing agency. The auditing agency prepares a PT report and sends a copy of the results to the Program Manager, Program QA Coordinator, and the EPA QA Office(s). Any results outside the acceptance criteria are noted in the PT report. Repeated analyte failures are investigated to determine the root cause and documented on a CAR. The PT reports are filed in the QA/QC file in Room 102. The performance on these audits is discussed in the annual QA Management Systems Review.

Currently, there is one audit program supported by this contract. This is provided through the NATTS program for carbonyl, metals, VOC, and PAH audits. These audits are provided to ERG from EPA (or an EPA contractor) throughout the year. The acceptable limits are provided on the annual reports presented to the participating States and EPA.

ERG participates in round robin studies, such as Regional EPA round robin studies, when available for VOC, metals, carbonyls, and SNMOC. In these studies, each participating laboratory result is compared against the calculated average. Reports from these studies are kept in the QA/QC file in Room 102. The performance on these studies is discussed in the annual QA Management Systems Review.

16.1.4 Data Assessment for Final Report

A data quality assessment is the statistical analysis of environmental data to determine whether the quality of data is of adequate quality, based on the MQOs. The data assessment in the final report is presented to EPA and State agencies and includes the following:

- Review of the MQOs of the program, which includes completeness, precision and accuracy.
- Present the results of the data quality assessment using summary statistics, plots and graphs while looking for and discussing any patterns, relationships, or anomalies.
- Qualify the data that does not meet the MQO for completeness for each monitoring site and for site-specific summary statistics.

16.2 Documentation of Assessments

16.2.1 TSA, Data Quality Audit, and PT Documentation

All reports from EPA or designated contractors regarding ERG's performance on TSAs, Data Quality Audits, and PTs are filed in the QA/QC file in Room 102. PT reports are dispersed and discussed with contributing staff.

Reports from internal TSAs are prepared and discussed with the contributing staff and Program Manager and filed in the QA/QC file in Room 102.

16.2.2 Internal Data Review Documentation

Internal data review is performed on 100 percent of the data by the Task Leader and 10 percent of the data by the Program QA Coordinator or designee against the criteria in the individual SOPs and this QAPP prior to being reported each month. The assessment is documented on the data review checklist, which is returned to the Task Leader for minor correction action and inclusion in the data package. The checklists used for analyses are shown in their respective SOPs (Appendix C) as follows:

- **VOC** – ERG-MOR-005, *SOP for the Concurrent GC/FID/MS Analysis of Canister Air Toxic Samples using EPA Compendium Method TO-15 and EPA Ozone Precursor Method.*

- **Carbonyl** – ERG-MOR-024, *SOP for Preparing, Extracting, and Analyzing DNPH Carbonyl Cartridges by Method TO-11A.*

- **SVOC/PAH** – ERG-MOR-049, *SOP for Analysis of Semivolatile Organic Compounds (Polynuclear Aromatic Hydrocarbons) Using EPA Compendium Method TO-13A & ASTM D6209.*

- **Metals** – ERG-MOR-095, *SOP for the Analysis of High Volume Quartz, Glass Fiber Filters, and 47 mm Filters for Metals by ICP-MS using Method IO 3.5 and FEM Method EQL-0512-201* and *FEM Method EQL-0512-202.*

- **Hexavalent chromium** – ERG-MOR-063, *SOP for the Preparation and Analysis of Ambient Air for Hexavalent Chromium by Ion Chromatography.*

- **SNMOC** – ERG-MOR-005, *SOP for the Concurrent GC/FID/MS Analysis of Canister Air Toxic Samples using EPA Compendium Method TO-15 and EPA Ozone Precursor Method.*

During the internal data review, major QC problems identified are brought to the attention of the Program Manager and are documented on a CAR. The final project report also addresses QA considerations for the whole project.

16.3 Corrective Action

The Response/Corrective Action Report (CAR) will be filed whenever a problem is found such as an operational problem, or a failure to comply with procedures that affects the quality of the data. A CAR is an important ongoing report to management because it documents primary QA activities and provides valuable records of QA actions. A CAR can be originated by anyone on the project but must be sent to the Program QA Coordinator and Program Manager. Any problem that affects the quality of the overall program will be discussed with EPA.

On the numbered CAR, the description of the problem, the cause of the problem, the corrective action, and the follow-up are documented. The follow-up assists the QA coordinator in determining if the corrective action was successful and if it was handled in a timely manner. The CAR is recorded on a three-part form, the white copy goes into the project file, the yellow copy goes into the QA file (Room 102), and the pink copy goes to the facilitator. A copy of the ERG CAR Form is shown in Figure 16-1.

Each recommendation addresses a specific problem or deficiency and requires a written response from the responsible party. The Program QA Coordinator will verify that the corrective action has been implemented. A summary of the past years' CARs are discussed during the annual QA Management Systems Review.

The following actions are taken by the laboratory QA Coordinator and Program Manager when any aspect of the testing work, or the results of this work, does not conform to the requirements of the quality system or testing methods:

- Identify nonconforming work and take actions such as halting of work or withholding test reports;
- Evaluate of the impact of nonconforming work on quality and operations;
- Take remedial action and make decision about the acceptability of the nonconforming work (resample, use as is with qualification, or unable to use);
- Notify the client, and if necessary, recall the work; and
- Authorize the continuation of work.

ERG and its subcontractors are responsible for implementing the analytical phase of this program and are not responsible for the overall DQOs. Therefore, this QAPP tries to ensure that analytical results are of known and adequate quality to ensure the achievement of the various program DQOs.

CAR Number: 2018-

ERG Corrective Action Report

CAR Initiator: Initiation Date:

Area/Procedure Affected: Click or tap here to enter text.

Is Immediate Stop of Work Required? Choose an item.

Non-Conformance

Date of Discovery:

Description of Non-Conformance: What happened? How is this a non-conforming event?

Click or tap here to enter text.

Investigation of Non-Conformance: How was the non-conformance discovered?

Click or tap here to enter text.

Impact Assessment: What is affected by the nonconformance?

Click or tap here to enter text.

Root Cause Analysis: What caused the nonconformance?

Click or tap here to enter text.

Further Analysis: Could this nonconformance be evident in other areas?

Click or tap here to enter text.

Corrective Action

Due Date for Remedial Action Completion:

Immediate and/or Long-Term Remedial Corrective Actions Taken:

Assessment of Corrective Action Effectiveness:

Click or tap here to enter text.

Signatures

	Signature & Date	Comments
QA Officer:		Click or tap here to enter text.
Project Manager:		Click or tap here to enter text.
Initiator:		Click or tap here to enter text.

Follow-up

Reference or attach documentation that demonstrates the return to conformance, or describe below.

Click or tap here to enter text.

Follow-up Auditor: Click or tap here to enter text. Date Completed:

Were corrective action procedures effective?

Click or tap here to enter text.

Figure 16-1. ERG Response/Corrective Action Report Form

SECTION 17
REPORTS TO MANAGEMENT

This section describes the quality-related reports and communications to management necessary to support monitoring network operations and the associated data acquisition, validation, assessment, and reporting. Important benefits of regular monthly reports to EPA provide the opportunity to alert of data quality problems, to propose viable solutions to problems, and to procure necessary additional resources.

Effective communication among all personnel is an integral part of a quality system. Regular, planned quality reporting provides a means for tracking the following:

- Adherence to scheduled delivery of data and reports;
- Documentation of deviations from approved QA and test plans, and the impact of these deviations on data quality; and
- Analysis of the potential uncertainties in decisions based on the data.

17.1 Frequency, Content, and Distribution of Reports

Frequency, content, and distribution of reports for monitoring are shown below.

17.1.1 Monthly and Annual Reports

Analytical data reports prepared by the Program or Deputy Program Manager are sent to EPA, State, Local and Tribal agencies monthly. These reports include the analytical data for each sample collected monthly including sample results, MDLs, sample information (canister ID, sample volume, etc.) and a QA report (could include duplicates, MB, CCB, CCV, MS/MSD, etc., depending on the analysis). Quarterly QA reports are distributed which include a summary of analyte specific quality control charts (ICV, ICB, CCB, CCV, BLK, BS/BSD, etc.). An annual data report, containing a summary of the monthly reported data and a yearly assessment of the air toxics data, is reported to EPA and State agencies by the Program Manager. This report documents the statistical analysis and quality assessment for the measurement data and how the objectives for the program were met.

The annual report includes the quality information for each toxic monitoring network in each state. Each report includes:

- Program overview and update;
- Quality objectives for measurement data;
- Data quality assessment;
- Collocated and duplicate sampling estimates for precision and bias; and
- PTs that were performed during the study, if applicable.

17.1.2 Internal Technical System Audit Reports

The Program QA Coordinator or designee performs an internal technical system audit at least once a year for the monitoring network for EPA, State, and NATTS contracts. The findings are listed in reports which are presented to the Program Manager and filed in the QA/QC storage file cabinet located in Room 102. These reports are available to EPA personnel during their TSA. More detail on internal TSAs is provided in Section 16.

DATA VALIDATION AND USABILITY

SECTION 18

DATA REVIEW AND VERIFICATION

Data verification is a two-stage process to determine if the sampling and analytical data collection process is complete, consistent with the DQOs discussed in this QAPP and associated SOPs, and meets the program requirements. First the data is reviewed for completeness, accuracy, and acceptability. Then the data is verified to meet the quality requirements of the program.

18.1 Data Review Design

Information used to verify air toxics data, includes:

- Sample COCs, holding times, preservation methods.
- Multi-point calibrations – the multipoint calibrations are used to establish proper initial calibration and can be used to show changes in instrument response.
- Standards – certifications, identification, expiration dates.
- Instrument logs – all activities and samples analyzed are entered into the LIMS logs (batches, sequences, etc.) to track the samples throughout the measurements procedures.
- Supporting equipment – identification, certifications, calibration, if needed.
- Blank, CCVs, replicate and spike results – these QC indicators can be used to ascertain whether sample handling or analysis is causing bias in the data set.
- Review Checklists – these record data quality review performed on all data by Task Leader and on 10 percent of the data by the QA Coordinator or designee. The checklists used to review data is presented in the SOPs.
- Summary Reports – monthly summary data reports present the preliminary data to EPA and respective State/Local/Tribal representatives including data qualifiers.

The reliability and acceptability of environmental analytical information depends on the rigorous completion of all the requirements outlined in the QA/QC protocol. During data analysis and validation, data are filtered and accepted or rejected based on the set of QC criteria listed in the individual SOPs included in Appendix C.

The data are critically reviewed to locate and isolate spurious values. A spurious value, when located, is not immediately rejected. All questionable data, whether rejected or not, are maintained along with rejection criteria and any possible explanation. Such a detailed approach can be time-consuming but can also be helpful in identifying sources of error and, in the long run, save time by reducing the number of outliers.

18.2 Data Verification

Data verification by examination confirms that specified method requirements have been fulfilled. The specific requirements are QC checks, acceptable data entry limits, etc. as presented in Section 11. The analytical procedures performed during the monitoring program will be checked against those described in the QAPP and the SOPs for the UATMP, PAMS, and NMOC support included in Appendix C. Deviations from the QAPP will be classified as acceptable or unacceptable, and critical or noncritical. During review and assessment, qualifiers will be applied to the data as needed; data found to have critical flaws (such as low spike for surrogate recoveries, contaminated blanks, etc.) will be invalidated and a CAR filled out and implemented, if needed. All data management guidelines followed for this contract are presented in Section 15.

18.3 Data Review

The COC forms are checked to ensure accurate transcription. The data are scrutinized daily to eliminate the collection of invalid data. The analyst records any unusual circumstances during analysis (e.g., power loss or fluctuations, temporary leaks or adjustments, operator error) on the LIMS bench sheet and notifies the analytical Task Leader.

QC samples and procedures performed during the monitoring program will be checked against those described in Section 11 of the QAPP. If QC is found unacceptable, corrective actions are implemented (as described in the same section). Prior to reporting, 100 percent of the data is reviewed by the Task Leader(s). To verify accuracy, at least 10 percent of the database is checked by the QA Coordinator or designated reviewer. Items checked can include original data sheets, checks of all calculations (from calibration to sample analysis), and data transfers. As the data are checked, corrections are made to the database as errors or omissions are encountered. If major errors are found, a greater percent of the data is checked to verify data quality. The Program Manager reviews all data before it is reported to EPA or the State/Local/Tribal agencies.

18.4 Data Reduction and Reporting

Monthly site-specific data summaries for the NMP are distributed to the participating EPA technical staff, administrators, and to the administrators of the State/Local/Tribal agencies involved in the study. NATTS, CSATAM, and UATMP data consists of any toxics including VOC, SNMOC, carbonyl, or other HAPs (metals, semivolatiles, etc.) requested by the program participants. Each report is prepared after 45 days from the end of the sampling month. Cumulative listings are periodically generated upon request. This timely turnaround of data assists in planning, preliminary modeling, and program development for the participating State/Local/Tribal agencies. Any changes made in the preliminary data because of subsequent data validation processes performed by EPA and/or State/Local/Tribal agencies are noted in the cumulative project data summaries for each specific sampling site. The data summaries include:

- Site code;
- Sample identifications;
- Sample dates;
- Target compound list;
- Concentrations (ppbv, ppbC, ng/m^3 and/or µg/m^3); and

- Method detection limits.

Preliminary monthly data summaries are emailed to the program participants. These data summaries are considered preliminary until the data is validated and entered into the AQS database, as detailed in Section 18.6.

The Program Manager reviews all data before they are reported to EPA and/or the State/Local/Tribal agencies. ERG prepares a final report containing all aspects of the individual programs including data summaries, QA, QC, and data analysis results for EPA, and distributes site-specific summaries of the final data to designated personnel.

18.5 Data Validation

Data validation is confirmed by examination of objective evidence that the requirements for a specific intended use are fulfilled as presented in Section 4. Intended use deals with data of acceptable quality to permit making decisions at the correct level of confidence. This data validation is performed prior to the annual final report. The data reported monthly are considered preliminary until the data is validated, entered into the AQS database, and reported in the annual final report.

The Precision from analysis of replicate samples in CV is determined by site, by compound, and as an average for the method. These precisions are based on analytical analyses only. Precision from the analysis and collection of duplicate/collocate samples in CV is determined by site, by compound, and as an average for the method. These precisions are based on analytical precision and sampling precision. The method average precision also includes collocated samples which can increase precision results. This measure the complete data set is compared against the data quality objective for the NATTS program, even though the other programs are not as stringent. This is accomplished prior to the preparation of the annual final report.

Representativeness can be assessed with site location information and is based on potential sources and select weather station information. This is accomplished while preparing the annual final report. Comparability is based on method measure of the level of confidence with which one data set can be compared to another. Ongoing data review and adherence to the data quality objectives keeps the data quality consistent and therefore comparable over the project. This is an ongoing data quality review followed by a data assessment prior to the preparation of the annual final report.

Completeness is measured by the amount of valid sample data obtained compared to what was expected. This is determined by counting the number of valid samples based on the sampling schedule for a that site. Eighty-five percent is considered complete for all the programs. This is an ongoing assessment used to facilitate make-up sampling in the same quarter when possible.

To ensure that the data is reliable in the ranges of concern, the minimum detection limit targets are those specified for the NATTS program, even though the other programs are less stringent. This is an ongoing assessment since detection limits are determined annually.

18.6 Air Quality System

ERG submits data collected for the NMOC, UATMP, NATTS, CSATAM, PAMS, and other air toxics programs to the AQS database.

Prior to ERG's submittal of data to AQS, the State/Local/Tribal agency submits, at a minimum, Basic Site Information transactions (Type AA) for each sampling site, and Site Street Information (Type AB) and Site Open Path Information (Type AC), if necessary. ERG then submits monitor transactions (Types MA through MN, as applicable) to prepare the AQS database for data upload. Data that are uploaded into AQS include Raw Data transactions (Type RD), QA transactions (Type Duplicate and Replicate, and Pb Analysis Audit) and Blank transactions (Type RB). ERG follows the NATTS TAD[18] to code data for the AQS database.

The submittal process involves the following steps:

- The raw data are formatted into pipe-delimited (|) coding that is accepted by AQS. Raw data, data generated by single sample episodes, by the primary sample (D1) of a duplicate episode, or by collocates (C1 and C2), are submitted using RD transactions. Precision data, data generated by Duplicate and Replicate samples (R1, D2, and/or R2), are submitted using QA transactions, specifically Duplicate and Replicate transactions. Accuracy data, generated for lead-FEM audit results, are also submitted using QA transactions.

- The RD QA (specifically duplicate, replicate and Pb Analysis Audit), and RB coding is generated and reviewed following guidelines specified in the *SOP for the Preparation of Monitoring Data for AQS Upload (ERG-MOR-098)* to ensure that the proper monitor ID (including state, county, site, parameter, and Parameter Occurrence Code (POC) codes), sampling interval, units, method, sample date, start time, and sample values are correct. The transactions are stored as text files for upload into the AQS database.

- Transaction files are primarily loaded under the Monitoring and Quality Assurance screening group.

- Transactions are edited to correct any errors found by AQS and then resubmitted. This step is repeated until the transactions are free of errors.

- AQS performs a statistical check on the data submitted to validate the data and determines if there are any outliers based on past data.

- Raw data (RD) transactions are then posted into the AQS database.

18.6.1 AQS Flagging and Reporting

Air toxics data submittals may be submitted with flags to indicate additional information related to the sample. There are two qualifier flag types that may be applied: Null codes and Qualifier codes.

- **Null Code** — assigned when a scheduled sample is not usable (e.g., canister leaked, canister damaged in shipment, etc.).

- **Qualifier Code** — used to note a procedural or quality assurance issue that could possibly affect the concentration of the value or the uncertainty of the result. These flags can also be applied to indicate atypical field conditions (e.g., nearby fires, construction in the area).

Qualifier Codes can be used in combination, with up to 10 possible codes applied. If a Null code is used, no other flag should be used since no results are reported. Table 18-1 presents the Qualifier codes and Table 18-2 presents the Null codes available to AQS users. These flags are applicable to the various steps of sample collection and analysis such as field operations, chain of custody, and laboratory operations.

Blank issue flags are qualifier flags used if reported blank values are above the limits set by the method SOPs or QAPP. If high blank values are associated with samples, the sample values are reported but appropriately flagged as described in the NATTS TAD[18]. Samples will not be invalidated due to high blank values. Blank issue flags are included in Table 18-1.

Table 18-1
Qualifier Codes

Qualifier Code	Qualifier Description
1	Deviation from a CFR/Critical Criteria Requirement
1V	Data reviewed and validated
2	Operational Deviation
3	Field Issue
4	Lab Issue
5	Outlier
6	QAPP Issue
7	Below Lowest Calibration Level
9	Negative value detected - zero reported
CB	Values have been Blank Corrected
CC	Clean Canister Residue
CL	Surrogate Recoveries Outside Control Limits
DI	Sample was diluted for analysis
DN	DNPH peak less than NATTS TAD requirement, reported value should be considered an estimate
EH	Estimated; Exceeds Upper Range
FB	Field Blank Value Above Acceptable Limit
FX	Filter Integrity Issue
HT	Sample pick-up hold time exceeded
IA	African Dust
IB	Asian Dust
IC	Chemical Spills & Industrial Accidents
ID	Cleanup After a Major Disaster
IE	Demolition
IF	Fire – Canadian
IG	Fire - Mexico/Central America

Table 18-1
Qualifier Codes, Continued

Qualifier Code	Qualifier Description
IH	Fireworks
II	High Pollen Count
IJ	High Winds
IK	Infrequent Large Gatherings
IL	Other
IM	Prescribed Fire
IN	Seismic Activity
IO	Stratospheric Ozone Intrusion
IP	Structural Fire
IQ	Terrorist Act
IR	Unique Traffic Disruption
IS	Volcanic Eruptions
IT	Wildfire-U. S.
J	Construction
LB	Lab blank value above acceptable limit
LJ	Identification of Analyte Is Acceptable; Reported Value Is an Estimate
LK	Analyte Identified; Reported Value May Be Biased High
LL	Analyte Identified; Reported Value May Be Biased Low
MD	Value less than MDL
MS	Value reported is ½ MDL substituted
MX	Matrix Effect
ND	No Value Detected, Zero Reported
NS	Influenced by nearby source
QP	Pressure Sensor Questionable
QT	Temperature Sensor Questionable
QX	Analyte does not meet QC criteria
SQ	Values Between SQL and MDL
SS	Value substituted from secondary monitor
SX	Does Not Meet Siting Criteria
TB	Trip Blank Value Above Acceptable Limit
TT	Transport Temperature is Out of Specs
V	Validated Value
VB	Value below normal; no reason to invalidate
W	Flow Rate Average out of Spec.
X	Filter Temperature Difference out of Spec.
Y	Elapsed Sample Time out of Spec.

Table 18-2
Null Codes

Null Code	Qualifier Description
AA	Sample Pressure out of Limits
AB	Technician Unavailable
AC	Construction/Repairs in Area
AD	Shelter Storm Damage
AE	Shelter Temperature Outside Limits
AF	Scheduled but not Collected
AG	Sample Time out of Limits
AH	Sample Flow Rate out of Limits
AI	Insufficient Data (cannot calculate)
AJ	Filter Damage
AK	Filter Leak
AL	Voided by Operator
AM	Miscellaneous Void
AN	Machine Malfunction
AO	Bad Weather
AP	Vandalism
AQ	Collection Error
AR	Lab Error
AS	Poor Quality Assurance Results
AT	Calibration
AU	Monitoring Waived
AV	Power Failure
AW	Wildlife Damage
AX	Precision Check
AY	Q C Control Points (zero/span)
AZ	Q C Audit
BA	Maintenance/Routine Repairs
BB	Unable to Reach Site
BC	Multi-point Calibration
BD	Auto Calibration
BE	Building/Site Repair
BF	Precision/Zero/Span
BG	Missing ozone data not likely to exceed level of standard
BH	Interference/co-elution/misidentification
BI	Lost or damaged in transit
BJ	Operator Error
BK	Site computer/data logger down
BL	QA Audit
BM	Accuracy check
BN	Sample Value Exceeds Media Limit
BR	Sample Value Below Acceptable Range
CS	Laboratory Calibration Standard
DA	Aberrant Data (Corrupt Files, Aberrant Chromatography, Spikes, Shifts)

Table 18-2
Null Codes (Continued)

Null Code	Qualifier Description
DL	Detection Limit Analyses
EC	Exceeds Critical Criteria
FI	Filter Inspection Flag
MB	Method Blank (Analytical)
MC	Module End Cap Missing
QV	Quality Control Multi-point Verification
SA	Storm Approaching
SC	Sampler Contamination
ST	Calibration Verification Standard
SV	Sample Volume out of Limits
TC	Component Check & Retention Time Standard
TS	Holding Time or Transport Temperature Is Out Of Specs.
XX	Experimental Data

ERG submits data to AQS using qualifier flags to show where the data are with respect to the detection level. A variety of terms and acronyms are used for defining the lowest level that can be detected for each analytical method. These terms and applications are derived from EPA's TAD for the NATTS program and are presented below:

- **Quantitation Limits (QL)** — the lowest level at which the entire analytical system must provide a recognizable signal and acceptable calibration point for the analyte.

- **Detection Limits (DL)** — the minimum concentration of an analyte that can be measured above instrument background.

- **MDL** — the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in each matrix containing the analyte (Part 136, App. B).

- **SQL** — the lowest concentration of an analyte reliably measured within specified limits of precision and accuracy during routine laboratory operating conditions. Normally, the SQL is determined as a multiplier of the method detection limit (e.g., 3.18 times) and is considered the lowest concentration that can be accurately measured, as opposed to just detected.

The qualifier flags associated with quantitation and detection limits are also included in Table 18-1, while Table 18-3 summarizes how they are applied to the data.

Table 18-3
Summary of Quantitation and Detection Limit Flags and Applications

If Concentration is:	Value to Report	Flag Applied
> SQL	Value	None
≥ MDL and ≤ SQL	Value	SQ
< MDL	Value	MD
Not Detected	0	ND

SECTION 19

DATA VALIDATION, VERIFICATION METHODS

Many of the processes for verifying and validating the measurement phases of the data collection operation have previously been discussed in Section 18. If these processes are followed, and the sites are representative of the boundary conditions for which they were selected, one would expect to achieve the DQOs. However, exceptional field events may occur, and field and laboratory activities may negatively affect the integrity of samples. In addition, it is expected that some of the QC checks will fail to meet the acceptance criteria. This section will outline how ERG will take the data to a higher level of quality analysis by performing software tests, plotting, and other methods of analysis.

19.1 Process for Validating and Verifying Data

19.1.1 Verification of Data

For the analytical data, the entries are reviewed to reduce the possibility of entry and transcription errors. Once the data are transferred to the ERG LIMS database, the data will be reviewed for routine data outliers and data outside acceptance criteria. These data will be flagged appropriately. Prior to reporting, 100 percent of the data is reviewed by the TL(s) and 10 percent of the database is checked by the QA Coordinator or designated reviewer. The PM also reviews the data prior to the preliminary report. After a preliminary reporting batch is completed, a review of 10 percent of the data will be conducted for completeness and manual and electronic data entry accuracy by the Annual Report/AQS TL.

19.1.2 Validation of Data

Data validation is performed by examination of objective evidence that the requirements for a specific intended use are fulfilled as presented in Section 4. Data is examined for representativeness, completeness, precision, and bias. This data validation, some of it performed with summary statistical analysis, is performed prior to the annual final report. Data validation is discussed in more detail in Section 18.5.

19.2 Data Analysis

Data analysis refers to the process of interpreting the data that are collected. Although there are a large number of parameters to analyze, many of these parameters present similar characteristics, (i.e., VOC, SVOC, and particulate metals, grouped according to their physical and chemical properties).

ERG will employ software programs, described below, to help analyze the data.

Spreadsheet – Select ERG employees perform analysis on the data sets using Excel® spreadsheets (analysts, Task Leaders, and QA reviewers) and Access® databases (AQS data entry). Spreadsheets and databases allow the user to input data and statistically analyze, graph linear data. This type of analysis will allow the user to see if there are any variations in the data sets. In addition, various statistical tests such as tests for linearity, slope, intercept, or correlation coefficient can be generated between two strings of data. Time series plots and control charts can help identify the following trends:

- Large jumps or dips in concentrations;
- Periodicity of peaks within a month or quarter; and
- Expected or unexpected relationships among species.

SECTION 20

RECONCILIATION WITH DATA QUALITY OBJECTIVES

The project management team, QA Coordinator, and sampling and analytical team members are responsible for ensuring that all measurement procedures are followed as specified and that measurements data meet the prescribed acceptance criteria. Prompt action is taken to correct any problem that may arise.

20.1 Conduct Preliminary Data Review

A preliminary data review will be performed as discussed in Sections 16 and 18 to uncover potential limitations to using the data, to reveal outliers, and generally to explore the basic structure of the data. The next step is to calculate basic summary statistics, generate graphical presentations of the data, and review these summary statistics and graphs to determine if the program requirements in Section 4, representativeness, comparability, completeness, precision, bias, and sensitivity, were met. Representativeness can be assessed with site location information and is based on potential sources and select weather station information. Comparability is based on method measure of the level of confidence with which one data set can be compared to another. Completeness is measured by the amount of valid sample data obtained compared to what was expected. Precision is determined from replicate analyses for a given method. Laboratory bias is demonstrated through PT samples and second source standards. Sensitivity is demonstrated through minimum detection limits.

20.2 Draw Conclusions from the Data

If the sampling design and statistical tests conducted during the final reporting process show results that meet acceptance criteria, it can be assumed that the network design and the uncertainty of the data are acceptable. This conclusion can then be reported to EPA and the States/Local/Tribal agencies, who then decide whether to perform risk assessments and analyze the data to determine whether these data can be used to address health effects.

SECTION 21

REFERENCES

1. McAllister, R. A., D-P. Dayton, and D. E. Wagoner. *1985 Nonmethane Organic Compounds Monitoring Assistance for Certain States in EPA Regions I, III, V, VI, and VII.* Radian Corporation, DCN No. 85-203-024-35-01, prepared for Dr. Harold G. Richter, Research Triangle Park, NC: U.S. Environmental Protection Agency, 1986.

2. *Technical Assistance Document for Sampling and Analysis of Ozone Precursors*. U.S. Environmental Protection Agency, National Exposure Research Laboratory, Research Triangle Park, NC. EPA 600-R-98/161. September 1998. Can be found at *https://www3.epa.gov/ttn/amtic/files/ambient/pams/newtad.pdf.*

3. Compendium Method TO-12, Determination of Non-Methane Organic Compounds (NMOC) in Ambient Air Using Cryogenic Pre-Concentration Direct Flame Ionization Detection (PDFID), 1999. Can be found at *https://www3.epa.gov/ttnamti1/files/ambient/airtox/to-12.pdf .*

4. Compendium Method TO-15, Determination of Volatile Organic Compounds (VOCs) In Air Collected In Specially-Prepared Canisters And Analyzed by Gas Chromatography/ Mass Spectrometry (GC/MS), 1999. Can be found at *https://www3.epa.gov/ttnamti1/files/ambient/airtox/to-15r.pdf.*

5. Compendium Method TO-11A, Determination of Formaldehyde in Ambient Air Using Adsorbent Cartridge Followed by High Performance Liquid Chromatography (HPLC), 1999. Can be found at *https://www3.epa.gov/ttnamti1/files/ambient/airtox/to-11ar.pdf.*

6. Compendium Method IO-3.5, The Determination of Metals in Ambient Particulate Matter Using Inductively Coupled Argon Plasma/Mass Spectrometry (ICP-MS), 1999. Can be found at *http://www.epa.gov/ttn/amtic/files/ambient/inorganic/mthd-3-5.pdf.*

7. EQL-0512-201, Standard Operating Procedure for Determination of Lead in TSP by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) with Hot Block Dilute Acid and Hydrogen Peroxide Filter Extraction, 2012. Can be found at *https://www3.epa.gov/ttn/amtic/files/ambient/pb/EQL-0512-201.pdf.*

8. EQL-0512-202, Standard Operating Procedure for the Determination of Lead in PM10 by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) with Hot Block Dilute Acid and Hydrogen Peroxide Filter Extraction, 2012. Can be found at *https://www3.epa.gov/ttn/amtic/files/ambient/pb/EQL-0512-202.pdf.*

9. ASTM D7614, Standard Test Method for Determination of Total Suspended Particulate (TSP) Hexavalent Chromium in Ambient Air Analyzed by Ion Chromatography (IC) and Spectrophotometric Measurements, 2012. Can be found at *https://www.astm.org/Standards/D7614.htm.*

10. Compendium Method TO-13A, Determination of Polycyclic Aromatic Hydrocarbons (PAHs) in Ambient Air Using Gas Chromatography/Mass Spectrometry (GC/MS), 1999. Can be found at *https://www3.epa.gov/ttnamti1/files/ambient/airtox/to-13arr.pdf.*

11. SW-846, Method 8270D, Semivolatile Organic Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS), 1996. Can be found at *http://www.epa.gov/epawaste/hazard/testmethods/sw846/pdfs/8270d.pdf.*

12. ASTM_D6209 Standard Test Method for Determination of Gaseous and Particulate Polycyclic Aromatic Hydrocarbons in Ambient Air (Collection on Sorbent-Backed Filters with Gas Chromatographic/Mass Spectrometric Analysis). Can be found at *https://www.astm.org/Standards/D6209.htm*.

13. Compendium Method TO-4A, The Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air Using High Volume Polyurethane Foam (PUF) Sampling Followed by Gas Chromatographic/Multi-Detector Detection (GC/MD), 1999. Can be found at *http://www.epa.gov/ttnamti1/files/ambient/airtox/to-4ar2r.pdf.*

14. NIOSH 7903, Acids, Inorganic, 1994. Can be found at *http://www.cdc.gov/niosh/docs/2003-154/pdfs/7903.pdf.*

15. Compendium Method TO-17, The Determination of Volatile Organic Compounds in Ambient Air Using Active Sampling Onto Sorbent Tubes, 1999. Can be found at *https://www3.epa.gov/ttnamti1/files/ambient/airtox/to-17r.pdf.*

16. OSHA Method 42, Diisocyanates (1,6-Mexamethylene Diisocyanate (HDI), Toluene-2,6-Diisocyanate (2,6-TDI), Toluene-2,4-Diisocyanate (2,4-TDI), 1989. Can be found at *http://www.osha.gov/dts/sltc/methods/organic/org042/org042.html.*

17. NIOSH Method 5029, 4,4′-Methylenedianiline, 1994. Can be found at *http://www.cdc.gov/niosh/docs/2003-154/pdfs/5029.pdf.*

18. *Technical Assistance Document for the National Air Toxics Trends Station Program.* U.S. Environmental Protection Agency. Office of Air Quality Planning and Standards, Research Triangle Park, NC, October 2016. Can be found at *https://www3.epa.gov/ttnamti1/files/ambient/airtox/NATTS%20TAD%20Revision%203_FINAL%20October%202016.pdf.*

19. U.S. Environmental Protection Agency. Code of Federal Regulations. Title 40, Chapter 1, Part 136, Appendix B. Office of the Federal Register, July 1, 1987. Can be found at *https://www.ecfr.gov/cgi-bin/text-idx?SID=dfbcc3c558942b0766bc1dba02b71d72&mc=true&node=ap40.25.136_17.b&rgn=div9.*

20. U.S. Environmental Protection Agency. Federal Advisory Committee Act (FACA). Can be found at *http://www.epa.gov/waterscience/methods/det/*.

Appendix A

ERG Exemptions from the NATTS TAD, Revision 3 & 4

2017 Quality Assurance Project Plan, Category 1
UATMP, NATTS, CSATAM, PAMS, and NMOC Support
(Contract No. EP-D-14-030)

The proposed **ERG EXEMPTIONS FROM THE NATTS TAD, REVISION 3,** listed in Appendix A of the QAPP have been deemed acceptable as noted by the signatures below.

Approved by:

U.S. EPA QA Manager: _______________ Date: 9/22/17

U.S. EPA Delivery Order Manager: _______________ Date: 9/30/17

ERG Program Manager: Julie L. Swift Date: 9/22/17

ERG Deputy Program Manager: Laura VanEnwyck Date: 9/22/17

ERG Program QA Officer: Donna Tedder Date: 9/22/17

ERG EXEMPTIONS FROM THE NATTS TAD, REVISION 3 (2017 QAPP, Contract EP-D-14-030)

ERG has provided the documentation to demonstrate that ERG meets the standard, in the form of historical data and/or experimental study results. These exemptions from Revision 3 NATTS TAD were approved and will remain in effect throughout the current contract.

Analyte	TAD Reference Location *	QC Parameter	ERG Exception	EPA Approval/Decision Dave Shelow (EPA Delivery Order Manager) & Greg Noah (QA Manager)
VOCs	4.2.2, pg 66	Both sample results must be qualified when entered into AQS for instances in which collocated or duplicate samples fail precision specifications.	The precision tables do not allow flags. Flags will be uploaded into AQS as permitted.	Approved at June 2017 EPA/ERG meeting (June 23, 2017)
VOCs	4.2.4.1.1.1, pg 74	Canisters with leak rates > 0.1 psi/day must be removed from service and repaired.	ERG evacuates the canisters to ~25" Hg and measured again in seven days. Our acceptance criteria is <1" Hg (QAPP section 11.1). This more accurately mimics the vacuum of the canisters shipped to the field when there is greater potential of major leak affecting the sample concentration.	Approved at June 2017 EPA/ERG meeting (June 23, 2017)
VOCs	4.2.4.2.4, pg 77 Table 4.2-3, pg 93	States on canister per batch cleaned in Section 4.2.4.2.4. but in Table 4.2-3 it states that the canister chosen must represent no more than 10 total canisters.	ERG heated canister cleaning systems are 12-port systems. We propose to continue verifying cleanliness on one canister for each batch of 12. Historical data can be provided if needed.	Approved at June 2017 EPA/ERG meeting (June 23, 2017)
VOCs	4.2.6, pg 80	The recommended tolerance is a pressure change of ≤0.5 psia.	Because of the wide variety of sites, gauges, operators, ERG has created a spreadsheet to track the pressure differences between field and laboratory. If these values differ by historical differences > 3", the samples are invalidated	Approved at June 2017 EPA/ERG meeting (June 23, 2017)

ERG EXEMPTIONS FROM THE NATTS TAD, REVISION 3 (2017 QAPP, Contract EP-D-14-030)

ERG has provided the documentation to demonstrate that ERG meets the standard, in the form of historical data and/or experimental study results. These exemptions from Revision 3 NATTS TAD were approved and will remain in effect throughout the current contract.

Analyte	TAD Reference Location *	QC Parameter	ERG Exception	EPA Approval/Decision Dave Shelow (EPA Delivery Order Manager) & Greg Noah (QA Manager)
VOCs	4.2.8.5.2.2, pg 87 Table 4.2-3, pg 93	Analysis of swept carrier gas through the Preconcentrator to demonstrate the instrument is sufficiently clean to begin analysis (IB).	This is listed as a recommendation in Section 4.2.8.5.2.2 but as a requirement in Table 4.2-3. Because the samples are checked with the analysis of blank samples, ERG will analyze the IB only for trouble shooting purposes.	Approved at June 2017 EPA/ERG meeting (June 23, 2017)
Carbonyls	4.3.2, pg 97	The sample must be kept cold during shipment such that the temperature remains ≤ 4°C, and the temperature of the shipment must be determined upon receipt at the laboratory.	This requirement will be extremely difficult to achieve during summer months and is not required in Method TO-11A. The vendor does not ship the cartridges to the laboratory in coolers but the samples are shipped overnight with receipt in the laboratory Tuesday through Friday. ERG will conduct a summer study to determine the necessity of this requirement and present it to the EPA in 2017.	Study presented to the EPA on August 25, 2017 validating ERG's exemption. The exemption was approved at this meeting.
Carbonyls	4.3.9.4, pg 115 Table 4.3-4, pg 121	EMSB - For batch sizes of more than 20 field-collected cartridges, n such QC samples of each type must be added to the batch, where n = batch size / 20, and where n is rounded to the next highest integer.	ERG has previously only performed this type of extraction to see if there were problems in a new lot of solvents. Our procedure will perform this extraction once a month, in the first batch of samples prepared each month.	Approved at June 2017 EPA/ERG meeting (June 23, 2017)

ERG EXEMPTIONS FROM THE NATTS TAD, REVISION 3 (2017 QAPP, Contract EP-D-14-030)

ERG has provided the documentation to demonstrate that ERG meets the standard, in the form of historical data and/or experimental study results. These exemptions from Revision 3 NATTS TAD were approved and will remain in effect throughout the current contract.

Analyte	TAD Reference Location *	QC Parameter	ERG Exception	EPA Approval/Decision Dave Shelow (EPA Delivery Order Manager) & Greg Noah (QA Manager)
Carbonyls	4.3.9.5.2, pg 117	For positive identification, the RT of a derivatized carbonyl must be within three standard deviations (3s) or ± 2%, whichever is smaller, of its mean RT from the ICAL	ERG's Carbonyl software (Agilent®) allows a ±2.5% window, not ±2.0%, but will automatically check if compounds are outside of this window. ERG believes the automatic function is advantageous and will perform LC maintenance checks if the RT fall outside this RT window.	Approved at June 2017 EPA/ERG meeting (June 23, 2017)
Metals	4.4.5, pg 128	Field blank analysis must demonstrate all target elements < MDL.	ERG does not get filters from the same lot that are provided to the field for sampling. Our filters are purchased and we determine the MDLs based on the background in that particular lot. Because of the wide variety of filter lots coming in from the different sites, and until the manufacturers of the filters provide clean enough samples, the majority of the elements could potentially be flagged. ERG proposes to flag only those elements over 5xMDL in order to better accommodate the potential lot differences.	Approved at June 2017 EPA/ERG meeting (June 23, 2017)

ERG EXEMPTIONS FROM THE NATTS TAD, REVISION 3 (2017 QAPP, Contract EP-D-14-030)

ERG has provided the documentation to demonstrate that ERG meets the standard, in the form of historical data and/or experimental study results. These exemptions from Revision 3 NATTS TAD were approved and will remain in effect throughout the current contract.

Analyte	TAD Reference Location *	QC Parameter	ERG Exception	EPA Approval/Decision Dave Shelow (EPA Delivery Order Manager) & Greg Noah (QA Manager)
Metals	4.4.10.5, pg 137	RBS- spiked digestion solution only (no filter strip – ensures proper spike recovery without the filter matrix)	ERG will prepare Standard Reference Material samples (required by NAAQS lead) and perform Post Digestion Spike analysis to ensure proper spike recovery without the filter matrix, instead of preparing and analyzing the RBS.	Approved at June 2017 EPA/ERG meeting (June 23, 2017)
Metals	4.4.10.5.2.1, pg 139	Each filter strip must be accordion folded or coiled and placed into separate digestion vessels.	ERG does not use accordion folding for the QFF filters. The digestion procedure is detailed in SOP 084. Historical data for over 10 years show acceptable recoveries using this method. ERG proposes to keep current folding procedures in place.	Approved at June 2017 EPA/ERG meeting (June 23, 2017)
Metals	4.4.11.7.1, pg 142	Replicate analyses of the calibration standards must show %RSD ≤ 10%	ERG's lowest calibration point is at the LOQ concentration. Our standard practice is to have all cal points at %RSD ≤10%, but the low cal point at %RSD ≤20%. This standard uses the same concentrations as the Limit of Quantitation (LOQ) standard, which are near or less than that of the MDL, therefore an RSD ≤ 20 percent is acceptable.	Added text in QAPP Section 11.3.5, "Replicate analysis of the calibration standards must have an RSD ≤ 10 percent, except for the second calibration standard (CAL2). This standard uses the same concentrations as the Limit of Quantitation (LOQ) standard, which are near or less than that of the MDL, therefore an RSD ≤ 20 percent is acceptable." Approved at June 2017 EPA/ERG meeting (June 23, 2017)

ERG EXEMPTIONS FROM THE NATTS TAD, REVISION 3 (2017 QAPP, Contract EP-D-14-030)

ERG has provided the documentation to demonstrate that ERG meets the standard, in the form of historical data and/or experimental study results. These exemptions from Revision 3 NATTS TAD were approved and will remain in effect throughout the current contract.

Analyte	TAD Reference Location *	QC Parameter	ERG Exception	EPA Approval/Decision Dave Shelow (EPA Delivery Order Manager) & Greg Noah (QA Manager)
Metals	4.4.11.7.3, pg 143 4.4.11.7.6, pg 144 4.4.11.8, pg 145 Table 4.4-3	The ICB is again analyzed following the ICV; all element responses must be less than the laboratory's established MDLsp for MDLs determined via Section 4.1.3.1 or the portion of the MDL represented by s·K for MDLs determined via Section 4.1.3.2. Also for CCB, negative values, BLK1, and RB.	ERG references the MDL for the ICB, CCB, negative values, reagent blanks and method blanks, not the s * K. ERG does not believe there should be 2 different sets of criteria for instrument/batch QC. These are all < MDL.	Approved at June 2017 EPA/ERG meeting (June 23, 2017)
Metals	4.4.11.7.4, pg 143 Table 4.4-3, pg 147	ICSA - All target elements < MDLsp (refer to Section 4.1.3.1) or s·K (refer to Section 4.1.3.2) – may be subtracted for ICS A certificate of analysis	ERG's critieria is for the results to be within ±3 times LOQ from zero or from the stock standard. This allows us to take into account the background in the interference solution when present.	Approved at June 2017 EPA/ERG meeting (June 23, 2017)
Metals	4.4.9.5.1, pg 132 4.4.10.5.1, pg 137 Table 4.4-3, pg 148	LCS - Recovery within 80-120% of nominal for all target elements, Sb recovery 75-125%.	ERG does not currently flag Sb if it is over 80-120%. ERG will monitor Sb with control charts for 6 months or gather existing data to allow us to statistically determine reasonable acceptance criteria.	Historical control charts presented and it was decided to flag QC and sample data starting 11/1/17. Discussed at the September 2017 EPA/ERG meeting (September 22, 2017)
Metals	4.4.10.5.1, pg 137 Table 4.4-3, pg 148	MS/MSD - Recovery within 80-120% of the nominal spiked amount for all target elements, Sb recovery 75-125%.	ERG does not currently flag Sb if it is over 80-120%. ERG will monitor Sb with control charts for 6 months or gather existing data to allow us to statistically determine reasonable acceptance criteria.	Historical control charts presented and it was decided to flag QC and sample data starting 11/1/17. Discussed at the September 2017 EPA/ERG meeting (September 22, 2017)

ERG EXEMPTIONS FROM THE NATTS TAD, REVISION 3 (2017 QAPP, Contract EP-D-14-030)

ERG has provided the documentation to demonstrate that ERG meets the standard, in the form of historical data and/or experimental study results. These exemptions from Revision 3 NATTS TAD were approved and will remain in effect throughout the current contract.

Analyte	TAD Reference Location *	QC Parameter	ERG Exception	EPA Approval/Decision Dave Shelow (EPA Delivery Order Manager) & Greg Noah (QA Manager)
PAH	4.5.3, pg 152 Table 4.5-3	Lot Blank - Regardless of the source of materials or the specific cleaning procedures each agency adopts, the QFF and PUF/XAD-2/PUF present in cartridges must meet the batch blank acceptance criteria of < 10 ng each for all target compounds. One cartridge for each batch of 20 or fewer prepared cartridges	ERG's procedure has been to prepare one filter per preparation shipment day. Background contamination (even when precleaned before preparing cartridges by the laboratory) show targets > 10 ng per target compound. ERG's criteria is to flag only those compounds which have recoveries > 5x MDL. ERG will monitor 6 months of lot blank data to provide to the EPA to justify exemption.	Historical control charts presented and it was decided to allow a new exemption criteria to be less than the MDL starting 11/1/17. Discussed at the September 2017 EPA/ERG meeting (September 22, 2017)
PAH	4.5.3.3, pg 153	Field surrogates are added no sooner than two weeks prior to the scheduled sample collection date.	ERG will be unable to provide sites with an extra sample media on each sampling day (standard practice) if we are not allowed to have cartridges spiked no sooner than two weeks. This practice is not listed in TO-13A or the ASTM 6209. ERG will perform a study or gather existing data to determine how long the spiked surrogates are stable on the cartridges (up to 3 months) and present it to the EPA to justify exemption.	Study presented to the EPA on August 25, 2017 validating ERG's exemption. The exemption was approved at this meeting.

ERG EXEMPTIONS FROM THE NATTS TAD, REVISION 3 (2017 QAPP, Contract EP-D-14-030)

ERG has provided the documentation to demonstrate that ERG meets the standard, in the form of historical data and/or experimental study results. These exemptions from Revision 3 NATTS TAD were approved and will remain in effect throughout the current contract.

Analyte	TAD Reference Location *	QC Parameter	ERG Exception	EPA Approval/Decision Dave Shelow (EPA Delivery Order Manager) & Greg Noah (QA Manager)
PAH	4.5.4.1b, pg 154	Samples which are shipped overnight should be packed with sufficient cold packs or ice to ensure they arrive at the laboratory at ≤ 4°C.	This requirement will be extremely difficult to achieve during summer months. ERG will conduct a summer study to determine the necessity of this requirement and present it to the EPA in 2017.	Study presented to the EPA on August 25, 2017 validating ERG's exemption. The exemption was approved at this meeting.
PAH	4.5.5.5.2, pg 160	Tuning the MS. Table 4.5-2	ERG currently uses the version from 8270D Rev5 July 2014 version which is the updated tune table for where the TO-13A method originally lifted their tune criteria. It is our opinion the original table listed (in Table 4.5-2) was created for older machines with less capability. The 2014 revision gives the operator the ability to tune to the heavier masses and get better resolution on the complex compounds. ERG proposes to continue using the 8270D criteria.	Approved at June 2017 EPA/ERG meeting (June 23, 2017)
PAH	4.5.5.5.3, pg 161	An SB which is not fortified with IS must be analyzed just prior to calibration to ensure the instrument is sufficiently clean to continue analysis. Analysis of the SB must show all target compounds, IS, and surrogate compounds are not detected	Table 4.5-3 states that the SB must be analyzed before each DFTPP tune, Section 4.5.5.5.3 states before each calibration. ERG will analyze the SB prior to the ICAL which is required in our DQOs not to exceed 6 weeks.	Approved at June 2017 EPA/ERG meeting (June 23, 2017)

ERG EXEMPTIONS FROM THE NATTS TAD, REVISION 3 (2017 QAPP, Contract EP-D-14-030)

ERG has provided the documentation to demonstrate that ERG meets the standard, in the form of historical data and/or experimental study results. These exemptions from Revision 3 NATTS TAD were approved and will remain in effect throughout the current contract.

Analyte	TAD Reference Location *	QC Parameter	ERG Exception	EPA Approval/Decision Dave Shelow (EPA Delivery Order Manager) & Greg Noah (QA Manager)
PAH	4.5.5.5.3, pg 162	The RRTs of each surrogate or target compound across the ICAL are then averaged to determine the ICAL RRT. All RRTs must be within ± 0.06 RRT units of RRT.	ERG's VOC software (ChemStation) allows different time deltas for lower and upper time limits. For instance, the window for acenaphthylene is RT – 0.175 and RT + 0.25. The largest delta in the database is RT + 0.25, and it's used for several compounds. These windows for each compound are well within those required using the mean RRT. A table presenting RRTs to ERG's current procedure of tracking RT's is presented in Appendix B.	Approved at June 2017 EPA/ERG meeting (June 23, 2017)
All Analytes	VOC Table 7.1, pg 190 Carbonyl, 4.3.8.1.3, pg 110 Metals, 4.4.9.4.1 & 4.4.10.4.1, pg 131 & pg 137 PAH, 4.5.4.1, pg 154	The sampling period for all field samples collected should be 1380-1500 minutes (24±1 hour) starting and ending at midnight.	ERG has reported any sample that was 22-23 hours or 25-26 hours, but flagged them with a "Y" (Elapsed Sample Time out of Spec.). Anything greater than ±2 hours is invalidated.	Approved at June 2017 EPA/ERG meeting (June 23, 2017)

ERG EXEMPTIONS FROM THE NATTS TAD, REVISION 4 (2018 QAPP, Contract EP-D-14-030)

ERG has provided the documentation to demonstrate that ERG meets the standard, in the form of historical data and/or experimental study results. These exemptions from Revision 3 NATTS TAD were approved and will remain in effect throughout the current contract.

Analyte	TAD Reference Location *	QC Parameter	ERG Exception	EPA Approval/Decision Dave Shelow (EPA Delivery Order Manager) & Greg Noah (QA Manager)
VOCs	4.2.3.5.1, pg 71	The zero check is performed by simultaneously providing humidified (50 to 70% RH) hydrocarbon- and oxidant-free zero air (must meet the cleanliness criterion of < 0.2 ppbv or < 3x MDL, whichever is lower) or UHP nitrogen to the sampling unit for collection into a canister and to a separate reference canister connected directly to the supplied HCF zero air gas source.	For the compound acetonitrile, ERG will use the previous criteria from TAD, Rev 2 of <0.2 ppbv.	Approved at July 2018 EPA/ERG meeting (July 27, 2018)

Appendix B

2018 Sampling Schedule

2018 6-Day Sampling Calendar

January

Sun	Mon	Tue	Wed	Thu	Fri	Sat
	1	2	3	4	5	6
7	D	9	10	11	12	13
14	15	16	17	18	19	20
21	22	23	24	25	FB	27
28	29	30	31			

February

Sun	Mon	Tue	Wed	Thu	Fri	Sat
				1	2	3
4	5	6	M	8	9	10
11	12	13	14	15	16	17
18	19	20	21	22	23	24
FB	26	27	28			

March

Sun	Mon	Tue	Wed	Thu	Fri	Sat
				1	2	3
4	5	6	7	8	D	10
11	12	13	14	15	16	17
18	19	20	21	22	23	24
25	26	FB	28	29	30	31

April

Sun	Mon	Tue	Wed	Thu	Fri	Sat
1	2	3	4	5	6	7
M	9	10	11	12	13	14
15	16	17	18	19	20	21
22	23	24	25	FB	27	28
29	30					

May

Sun	Mon	Tue	Wed	Thu	Fri	Sat
		1	2	3	4	5
6	7	D	9	10	11	12
13	14	15	16	17	18	19
20	21	22	23	24	25	FB
27	28	29	30	31		

June

Sun	Mon	Tue	Wed	Thu	Fri	Sat
					1	2
3	4	5	6	M	8	9
10	11	12	13	14	15	16
17	18	19	20	21	22	23
24	FB	26	27	28	29	30

July

Sun	Mon	Tue	Wed	Thu	Fri	Sat
1	2	3	4	5	6	7
8	9	10	11	12	D	14
15	16	17	18	19	20	21
22	23	24	25	26	27	28
29	30	FB				

August

Sun	Mon	Tue	Wed	Thu	Fri	Sat
			1	2	3	4
5	6	7	8	9	10	11
M	13	14	15	16	17	18
19	20	21	22	23	24	25
26	27	28	29	FB	31	

September

Sun	Mon	Tue	Wed	Thu	Fri	Sat
						1
2	3	4	5	6	7	8
9	10	D	12	13	14	15
16	17	18	19	20	21	22
23	24	25	26	27	28	FB
30						

October

Sun	Mon	Tue	Wed	Thu	Fri	Sat
	1	2	3	4	5	6
7	8	9	10	M	12	13
14	15	16	17	18	19	20
21	22	23	24	25	26	27
28	FB	30	31			

November

Sun	Mon	Tue	Wed	Thu	Fri	Sat
				1	2	3
4	5	6	7	8	9	D
11	12	13	14	15	16	17
18	19	20	21	22	23	24
25	26	27	FB	29	30	

December

Sun	Mon	Tue	Wed	Thu	Fri	Sat
						1
2	3	4	5	6	7	8
9	M	11	12	13	14	15
16	17	18	19	20	21	22
23	24	25	26	27	FB	29
30	31					

Standard Sample Collection

FB Field Blank Collection

M Makeup Duplicate Collection or normal sample

D Duplicate Sampling Collection

Appendix C

Relevant ERG Standard Operating Procedures

Appendix D

Subcontractors

Quality Assurance Project Plan

RTI Laboratories

Will be provided when work is initiated.